



AN EVALUATION OF EFFLUENTS  
GENERATED FROM A THERMOMECHANICAL  
PULP MILL

NOVEMBER 1979

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Ontario

Ministry  
of the  
Environment

The Honourable  
Harry C. Parrott, D.D.S.,  
Minister

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Deputy Minister

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AN EVALUATION OF EFFLUENTS FROM A  
THERMOMECHANICAL PULPING PLANT

ABSTRACT

Effluents of the Thermomechanical Pulping (TMP) process were evaluated in a cooperative study carried out at a fully operational TMP plant in Ontario.

A two year study investigating effluents from a thermomechanical pulp (TMP) mill is outlined. The mill studied had the capability of segregating the TMP unit from other plant sections. Loadings of BOD<sub>5</sub> suspended solids, and dissolved solids in effluents were determined. A method of analysing resin and fatty acids was developed and used to determine the organic acid content of effluents. A water balance was prepared to assure that all effluent streams were accounted.

Resin acid concentrations in effluent samples were reduced by ion exchange treatment techniques. The relationship between lethality and organic acid concentrations was investigated.

#### SPONSORSHIP OF STUDY

The following report was prepared by a joint Federal/Provincial investigation group known as the Thermomechanical Pulping - Effluent Evaluation Committee.

This committee was formed in April 1977 under the auspices of the Ontario Ministry of the Environment. The committee conducted field study programs during the summers of 1977 and 1978. This report is a final report outlining the results obtained from the studies undertaken.

The 'Committee' is represented by staff from the Ontario Ministry of the Environment, Environment Canada and by the pulp and paper industry.

The objectives of the group have been to gain empirical data regarding effluents from an operational TMP mill. It is hoped that these investigations will assist interested parties in gaining an improved perspective of the characteristics of discharges from this relatively new process.

1.0 SUMMARY

This investigation afforded the opportunity of determining waste loads generated solely from a TMP mill, since other integrated whitewaters were not allowed to recirculate into the TMP unit.

A reproducible water balance for the TMP mill demonstrated an improved confidence in the data derived in the 1978 study.

BOD<sub>5</sub> loadings were found to be 27 kg/adt.

Dissolved solids loadings were found to be 37 kg/adt whereas Suspended Solids loadings were 20 kg/adt.

The analytical methods developed for determining resin and fatty acids were suitable for describing TMP wastes.

Organic acid concentrations increased significantly during the 1978 surveillance. The use of fresh chips in 1978 appeared to be partially responsible for this observation.

The recirculation of preheater plug screw feeder effluent was deemed to be a significant factor in the organic acid content present in chip washer effluents. That stream, however, is small in volume and may be amenable to treatment.

All TMP effluent streams from this operation were found to be toxic to fish. By adsorption with a polymeric resin the concentration of organic acids could be reduced resulting in a corresponding reduction in toxicity.

### INTRODUCTION

- 2.0 The installation of TMP mills within the Province of Ontario has occurred on a cautious note. The first TMP unit to operate commercially came on-line in July 1976.

At this location the company operates an integrated chemical and mechanical mill. Chemical furnish is provided by a calcium sulphite plant producing approximately 140 air dried tonnes per day (adt/d); and a magnefite plant producing about 130 adt/d. Mechanical fibre is provided by a 510 adt/d stone groundwood operation and the TMP unit which provides some 190 adt/d. Products are 900 adt/d of newsprint paper and 35 adt/d of market sulphite. This market pulp is sold to an adjacent tissue mill which produces approximately 60 adt/d.

The installation of the TMP unit at this mill provided the Ontario Ministry of the Environment (M.O.E.) the opportunity of a first-hand investigation of effluents from this relatively new process.

Discussions with the company began in early 1977 aimed at developing a program which would allow a thorough characterization of liquid effluents from TMP operations. A preliminary investigation involving the company, the Federal Government represented by Environment Canada (E.P.S.) and the Ontario Ministry of the Environment; was completed during the summer of 1977. The investigation was expanded with refined survey techniques during the summer of 1978.

The objectives of these studies were: (a) to determine a water balance for the TMP mill; (b) to determine waste loadings (kg/tonne) of BOD<sub>5</sub>, dissolved solids, suspended solids, resin and fatty acids, (c) to develop and confirm analytical procedures for the analysis of resin and fatty acids (d) to determine the acute fish toxicity of TMP effluents; (e) to attempt to correlate resin and fatty acid concentrations and toxicities; and (f) to observe any reductions in toxicity upon treatment of TMP effluents to remove organic acid components.

3.0 DESCRIPTION OF TMP MILL:

At the TMP mill surveyed black spruce chips (80%) are normally off-loaded from trucks and stored on a 4500 tonne pile. The chips are transferred pneumatically to a live bottom silo; from which they are fed into a rotating washer using process hot water. Chip washer reject solids are settled in a grit trap and discharged to the sewer. Excess wash water is also sewerred. Cleaned chips are fed via a screw feeder to a pressurized steam pre-heater. Effluents from the pre-heater and from the screw feeder are returned to the chip washer circuit.

Pre-steamed chips are primary refined in a pressurized 6,000 kw Defibrator refiner. Primary pulp and steam discharge from the unit, with steam being recovered for plant uses. Stock proceeds to a secondary Defibrator refiner operating under atmospheric pressure. Pulp from this refiner is stored in a soak chest to reduce latency. Stock is then centricleaned and cleaned; with usable low grade fibre from the centricleaners returned for additional secondary refining. Rejects from the last stage of centricleaning are sewerred. Finished pulp is disc filtered and stored in a high density tank. TMP furnish is pumped directly to an adjacent newsprint mill for use (Figure 1).

#### 4.0 METHODOLOGY OF STUDY

Ideally a TMP mill should have few discharges to sewer. Dissolved materials released during refining are incorporated mainly into the overall mill whitewater system.

Evaluating effluents from TMP units is complicated by the fact that TMP whitewaters are usually mixed with recycled process waters from paper mill and groundwood mill sections. Consequently a TMP unit often has an equilibrated load of dissolved materials present before any TMP processing begins. In the case of the TMP plant investigated, the mill could be operated using fresh water only with no recirculation of whitewater from paper mill/groundwood sections. 'Cross contamination' could thus be avoided from other process waters. During both the 1977 and 1978 studies integrated paper mill/groundwood mill whitewater was shut off approximately 36 hours prior to sampling. Therefore an inplant recirculating load of dissolved materials, generated solely from the TMP mill, was achieved.

Sampling proceeded during 1977 mainly to identify and quantitate contaminants generated from various sections of the TMP mill. Effluents were sampled on a 'grab' sample basis over a two day period.



Discharges from the chip washer and centricleaner rejects were sampled. Plant water and TMP white-water, acting as a medium to transport pulp to the paper mill, were sampled. Samples from all input/output sources were collected for bioassay testing. Organic acid samples were preserved by adjustment to pH3 using HCl. All chemical analysis were performed at the M.O.E. laboratory in Toronto. (Appendix I - 1977 Data).

The investigation during 1978 was designed to improve the reliability of the data which had been developed the previous summer. In order to determine the distribution of materials entering and leaving the TMP unit an accurate flow balance was required.(Figure 2).

Water entered the mill by three sources. Fresh water was pumped to a heater where it was flashed with process steam. Chips entering the mill contained a proportion of the input water. Steam entering the process, which condensed, was included as a water input. Liquids removed from the process were: TMP whitewater (to paper mill); effluents sewered from the chip washer; aqueous discharges to sewer from centricleaners; and miscellaneous clean up and gland water discharges to sewer.

The TMP mill utilized several flow measuring devices to monitor inputs of fresh water, steam, and chips. Measuring equipment also monitored pulp flow to the paper mill. A Parshall flume in a combined effluent sewer indicated sewer flows, which then proceeded to a primary treatment clarifier. Mill measuring equipment for incoming water and TMP whitewaters were calibrated by the survey team prior to use. A salt dilution technique was used by which reproducible 'correction factors' for mill instruments were established. Concentrated standard solutions of sodium chloride were injected into the streams for calibration (Figure 3). Samples were collected after suitable mixing and analysed by field staff using a flame photometer. Discharge flows from chip washing and centricleaning were determined by timing known volumes. Final combined effluent flows were recorded by a level recorder installed in a Parshall flume. These were substantiated manually by measuring the depth of the flow passing the throat of the flume. Water balance data was collected over a period of four consecutive days (Appendix II - Flow Calibration).

To accurately account for the weight of chips used during sampling the chip weightometer was calibrated. Chips in the storage silo were used until a certain reference level was reached. For a two-day period trucks arriving with chips were weighed routinely on a scale and the chips were then discharged into a storage silo. Weightometer readings at the start and end of the test period trial were recorded. Chip samples were taken from each load and their moisture content determined. The input of water from this source could thus be determined. A calibration factor was developed for the chip weightometer which was used throughout the study.

Steam input from external sources was minimal during the course of the study (400-500 kg/hr). Steam flow was noted from process monitoring equipment.

## 5.0 ANALYTICAL PROCEDURES

All moisture and pulp consistency analyses were performed by mill staff using methods outlined by Standard Methods, Technical Section CPPA. (1). Samples were analysed for BOD<sub>5</sub>, dissolved solids, suspended solids and pH according to M.O.E. standard procedures (2).

Bioassay tests in 1977 were conducted at a M.O.E./E.P.S. field laboratory near Sudbury. Test and control water was drawn from Lake Panache, a typical pre-Cambrian shield Lake, exhibiting low hardness and conductivity (pH 6.9). Toxicity testing during 1978 was completed at the Ministry laboratory in Toronto.

Effluent samples for the 1978 bioassay study were collected as composites during a one day period of the study; then shipped directly to the M.O.E. lab in Toronto. Ten rainbow trout (*Salmo gairdneri*) each weighing 5.0g were exposed to the effluents under investigation. Duplicate controls were established where possible. Solutions were aerated at 5-7.5 cc/min/l throughout the tests. Temperatures were maintained during these tests at 14-16°C. Fish loadings were approximately 1.4 l/g/day. Effluents were treated to remove resin and fatty acids.

Treatment involved passing the effluent through a porous polymer resin, Amberlite<sup>R</sup> X AD-7, as prescribed by previous researchers. (4).

Procedures were developed to analyse resin and fatty acids by gas chromatography (G.C.). Quantitative and qualitative analysis for six resin and nine fatty acids were developed during 1977. Two additional resin acid compounds; derivatives of abietic acid, were added to the technique during 1978. Phthalic acid, a fatty acid; as well as benzoic and salicylic acids were also incorporated into the G.C. technique during 1978.

The principle of the extraction technique is to take weakly acidified samples (pH3) and extract them in diethyl ether. The extract is separated, dried and concentrated to a small volume. By reaction with diazomethane, the organic acids present are converted to the corresponding esters. Using a gas chromatograph with a flame ionization detector and with temperature programming, the esters were separated on a column of SP216 Supelcoport (R). By relating peak heights and retention times of components in the samples, to those of organic acid standards, qualitative and quantitative estimations of the organic acids in the samples could be accomplished. Detection limits for resin and fatty acids were 0.02 mg/l and 0.05 mg/l, respectively.

Extraction recoveries were tested by using known quantities of resin and fatty acids in aqueous solutions. Extraction efficiencies were found to range between 88% and 95%. Figure 3 depicts a typical recorder printout illustrating the compounds normally observed. Dr. E.G. Adamek of the Ministry of the Environment laboratory in Toronto expects to publish further refinements of this procedure in the near future. (Figure 4) Appendix III - Organic Acid Analysis).

## 6.0 RESULTS AND DISCUSSIONS

Upon calibration of mill flow measuring equipment, water balances covering four days of plant operations were developed from instrument readings. During the course of the study, water accountability was almost 100% (Table 1) (Appendix IV - Water Balance).

Results from data collected in 1977 were developed based upon two conditions which could occur in handling the final pulp. The company tested the use of a brightening agent, sodium hydrosulphite. Effluents from the manufacturing of both brightened and un-brightened TMP pulp were investigated during the 1977 study.

BOD<sub>5</sub> determination during the 1977 study indicated a total generation rate of 20 kg/air dry tonne (adt). BOD<sub>5</sub> generation increased moderately (10-15%) upon addition of a brightening agent. Dissolved solids loadings totalled 33 kg/adt without brightening agent addition. Dissolved solids generation increased to 36 kg/adt when sodium hydrosulphite was added. The major source of discharge of dissolved materials is from the TMP whitewater representing about 80% of the total. Total resin acid discharge during the 1977 work was approximately 1 kg/adt, whereas fatty acid discharge was 0.1 kg/adt.

Fresh chips charged directly from trucks to the TMP mill were used during the 1978 field trials. Noticeable increases in various parameters were noted and may be partially attributed to chip history. (Figure 5).

Loadings of BOD<sub>5</sub> during the 1978 study were 27 kg/adt; whereas dissolved solids generation increased to 37 kg/adt. No brightening agents were added in 1978.

Total suspended solids discharges were observed to be 20 kg/adt during the course of the study. Table 2 highlights the results obtained throughout this study (Table 2) (Appendix V - 1978 Results).

The most significant difference between the 1977 - 1978 field studies was that the concentration of organic acids during the latter part of the study increased markedly. During the 1977 work, total resin acid discharges were 1.3 kg/adt corresponding to less than 250 kg/day. As Figure 6 illustrates, the discharge of resin acids during the 1978 survey rose to 3.6 kg/adt or 705 kg/day/ when fresh chips were processed. This increase was noticed proportionally for all measured streams within the TMP unit during the 1978 test. The majority of the organic acids released (70%) discharged predominantly (about 70%) from the mill as TMP whitewater.



The next most significant discharge of organic acids was from the chip washer circuit. This may reflect the recirculation of plug screw feeder effluent to this unit. (Table 3) (Figure 6).

The most predominant resin acids identified from TMP effluents were abietic, neoabietic and dehydroabietic acids; in order of increasing concentration. These acids accounted for approximately 90% of the resin acids present. Minor resin acid components were identified in order of concentration, as isopimaric, sandaracopimaric, levopimaric and pimaric acids.

In the case of fatty acids the only major compounds noted were linoleic and oleic acids. Benzoic acid was grouped with these acids and provided the major constituent reported as fatty acids (60%).

The discharge of resin and fatty acids by pulp mills to adjacent receiving waters has been viewed as a major factor in the observed toxicity of these wastes throughout the literature (5, 6). In fact, investigators have suggested that resin acid concentrations well below 10 mg/l have shown acute toxicity and,

to a lesser extent the same observations have been made for certain unsaturated fatty acids. (7) Wood supply and storage history has also been noted to be a contributing factor in organic acid content, hence to the toxicity (8).

All TMP effluents tested were found to fail the Environment Canada fish toxicity regulation criteria; with only the input process warm water being the exception. This federal regulation considers an effluent to be toxic, should less than 80% of the fish survive; in a test solution of 65% (v/v) (9). Test duration is a standard 96 hours (Appendix VI - Bioassay Results).

For TMP process wastes the  $LC_{50}$  concentrations ranged between 0.9 to 3.6% (v/v). Mean survival time data indicated that acute toxicity in 1978 may have been more pronounced than in the previous year, however data was not conclusive. Effluents from the process were extremely toxic in all cases and thus masked the subtle differences observed in toxicity between the 1977 and 1978 studies.

Effluent treatment studies, using a packed column containing Amberlite<sup>R</sup> XAD-7 (Rohm & Haas Co., Phil. P.A.), were performed with the objective of removing all or a portion of the organic acids present.

By treating samples of TMP chip washer effluent and centricleaner effluent, removal efficiencies were noted ranging in the 50 - 70% range in the case of resin acids; and from 40 - 80% range for fatty acids (Table 4). Removal of benzoic acid was almost complete. Treated effluents were then bioassayed in the same manner as the raw samples. Both chip washer and centricleaner effluents were rendered less toxic by this treatment (Figure 7).

Observed reduction of effluent lethality were approximately equivalent to the percent removal of resin acids by treatment (Figure 7, Figure 8) (Appendix VII - Treatment Studies).

## 7.0 CONCLUSIONS

This study of the TMP effluents was made possible by the cooperation of the company who arranged the segregation of the TMP mill from the rest of the pulp and paper mill complex during the test period. Since no process whitewaters were allowed to enter the TMP mill, a determination of the waste loading generated solely from that mill was possible.

The development of a reproducible water balance for the TMP mill improved the confidence of data in the 1978 portion of the study.

BOD<sub>5</sub> loading from TMP operations was found to be 25-30 kg/adt. Dissolved solids amounted to approximately 40 kg/adt. Suspended solids loads were 20 kg/adt. The analytical methods used to determine resin and fatty acids showed a high degree of recovery efficiency and appeared to be suitable for the characterization and quantification of these acids in TMP effluents. Confirmation of some of the organic acids by using spectrometry or other suitable methods is recommended.

Organic acid concentrations increased significantly in 1978 as compared to data developed in the previous year. The use of fresh chips (less than one week old) in 1978, in contrast to stockpiled chips in 1977, appeared to be partially responsible for this increase. Abietic, neoabietic and dehydroabietic acids were the predominant resin acids present in waste streams. In addition linoleic, oleic, and benzoic acids were other organic acid compounds which were identified in the effluent.

The recirculation of plug screw feeder effluent was suspected to be a factor contributing significantly to the accumulation of dissolved solids and organic acid concentrations of the chip washer effluent.

During the two year study all process effluent streams were found to be toxic in nature. The most toxic effluents were the chip washer discharge, followed by TMP whitewater and then the centricleaner rejects effluent. Process warm water was non-toxic during the period of this study.

By adsorption on a polymetic resin, portions of the organic acids present could be removed from the effluents. Removal efficiencies were low but permitted a comparison of toxicity between raw and treated TMP wastes. Reductions in toxicity occurred when organic acid concentrations were reduced.

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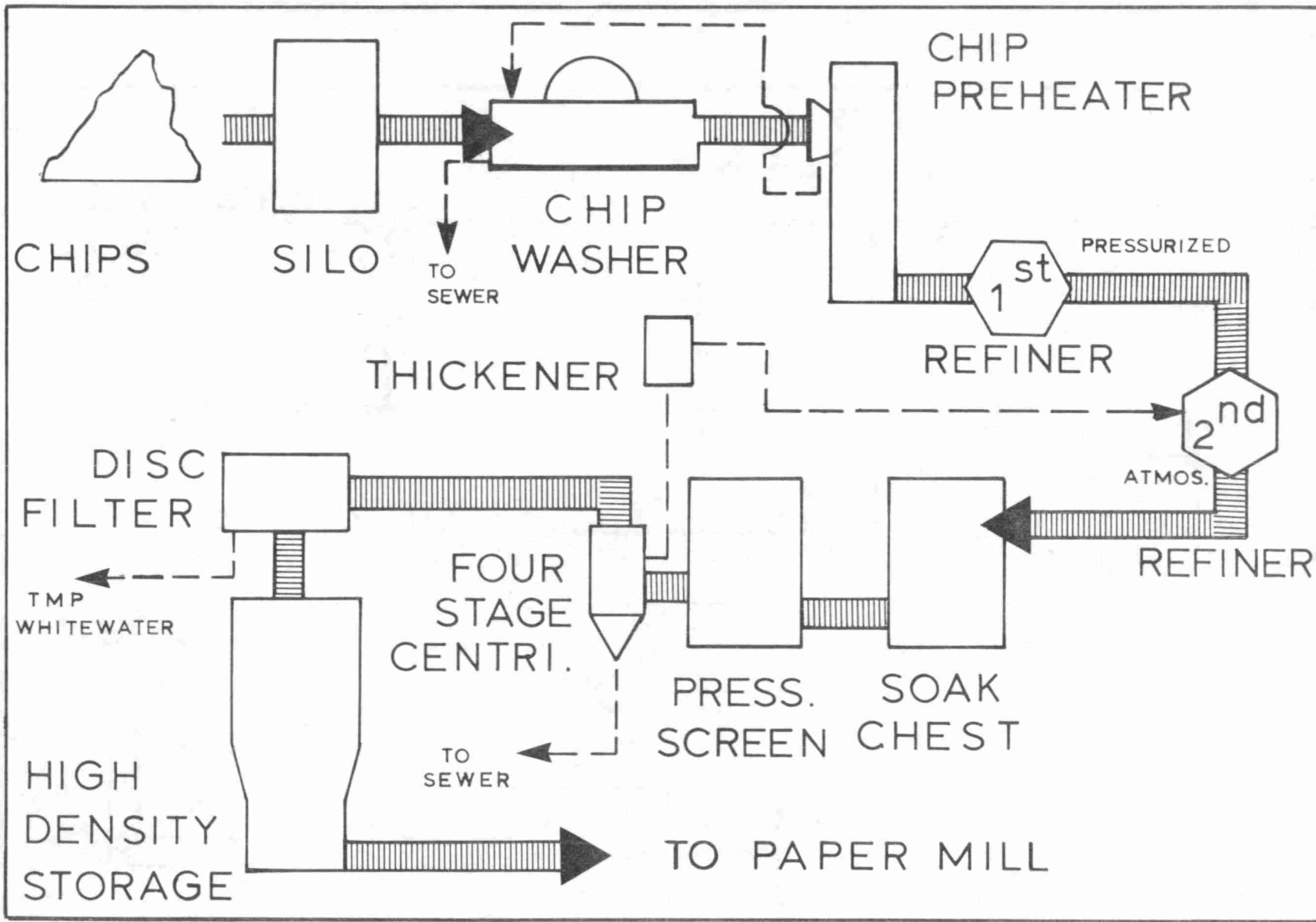
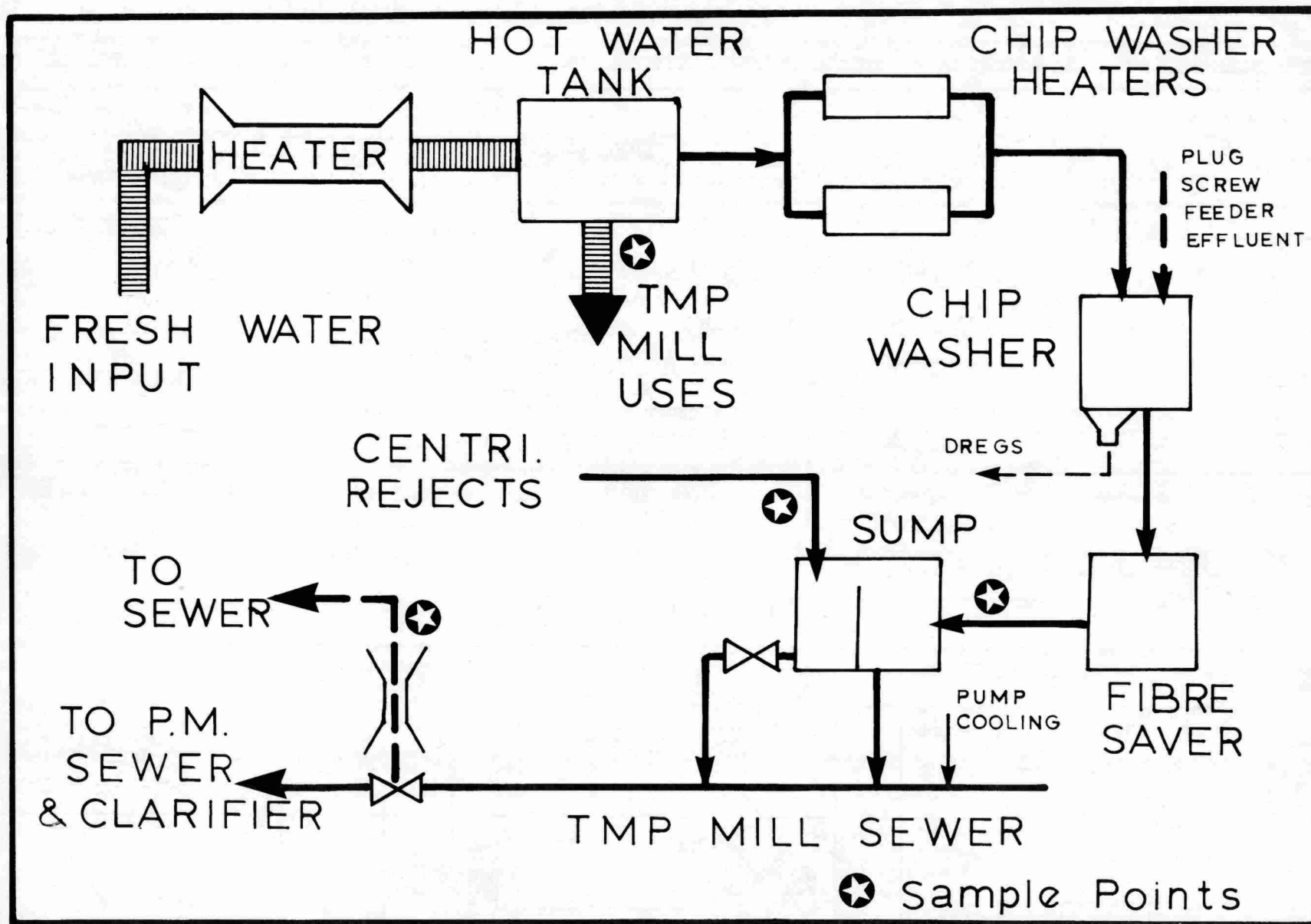


FIGURE 1

PROCESS FLOW SHEET



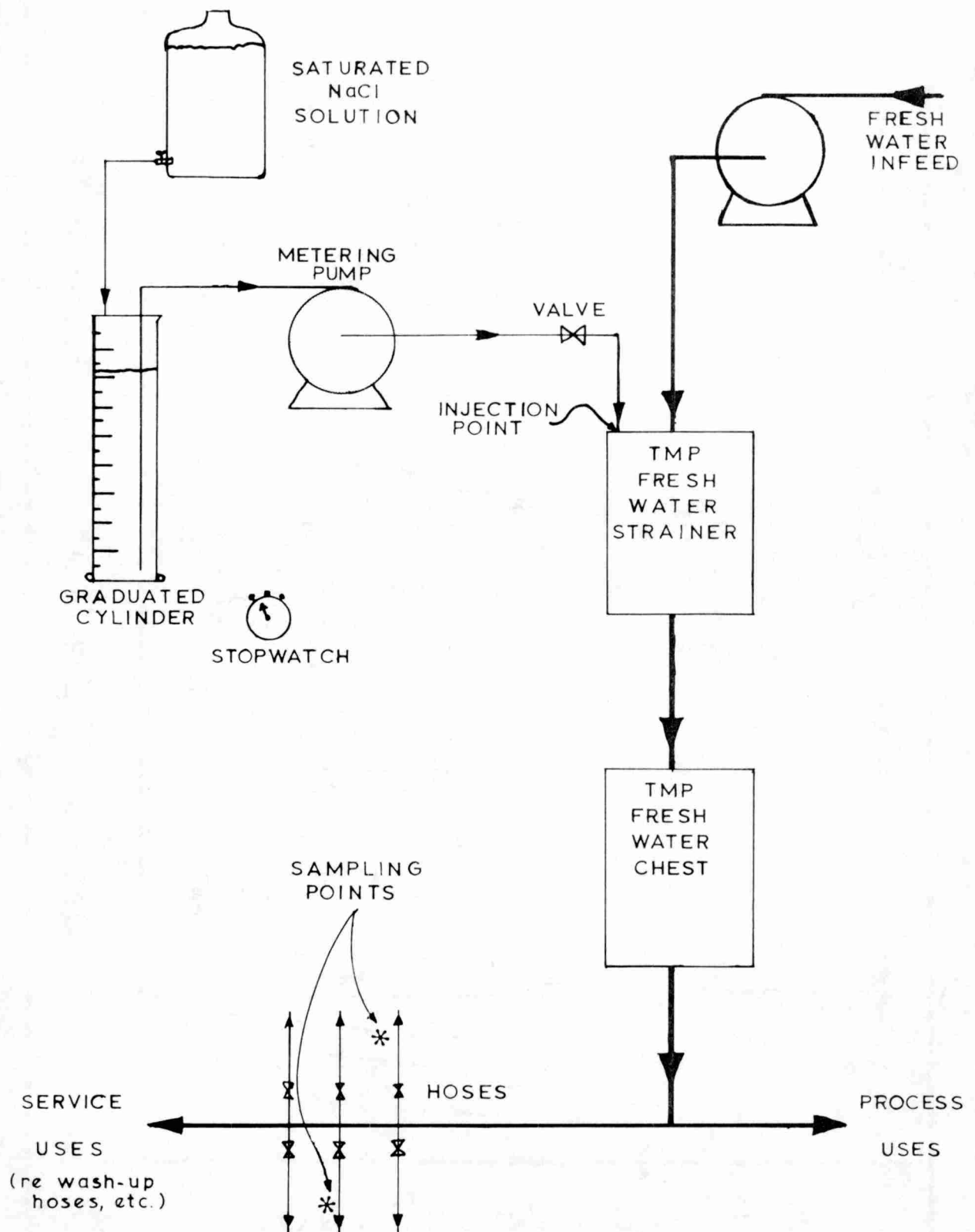
WATER & EFFLUENT FLOW

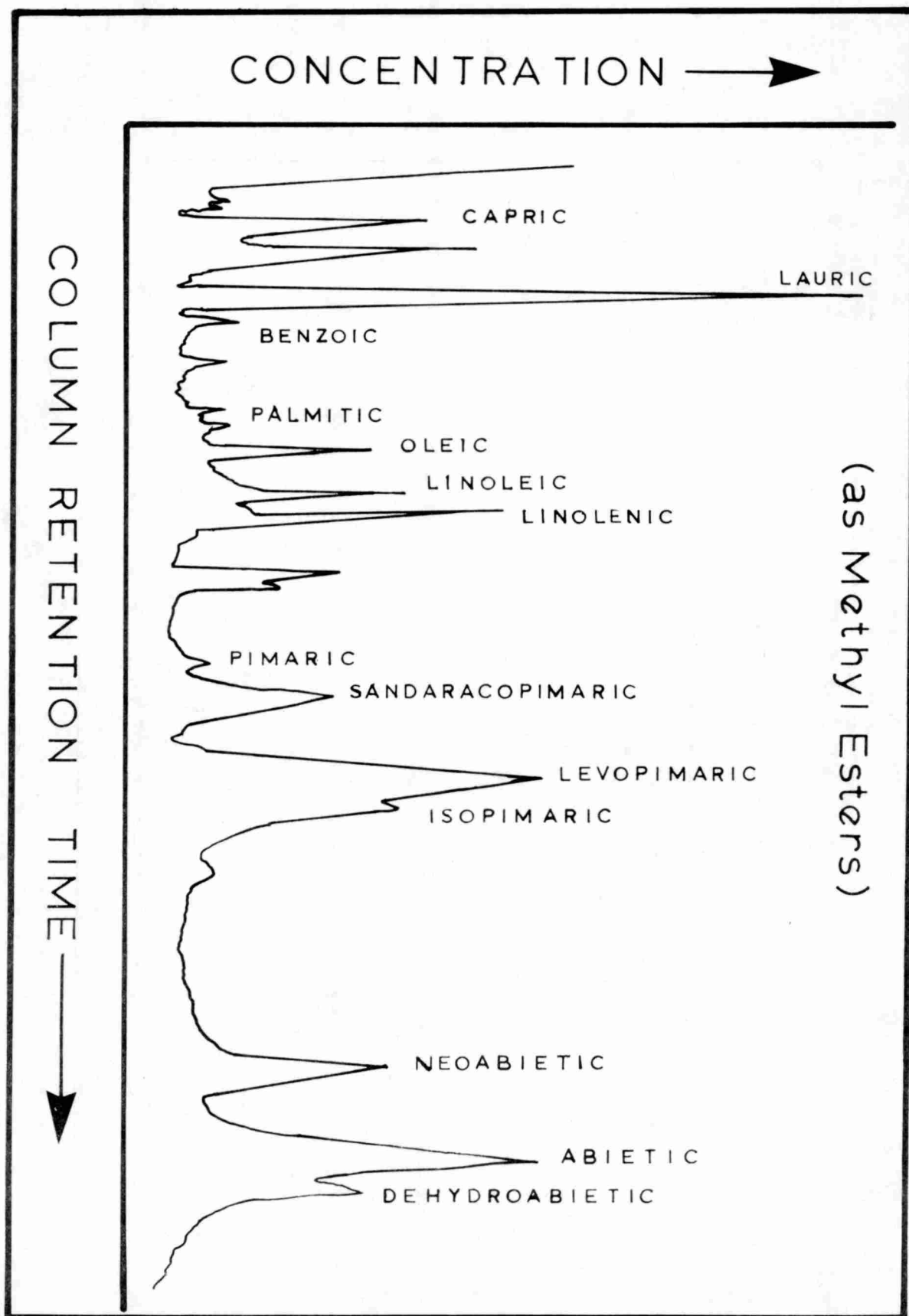
Ontario



# FIGURE 3

## TMP FRESH WATER CALIBRATION





CHROMATOGRAM

TYPICAL GAS

FIGURE 4



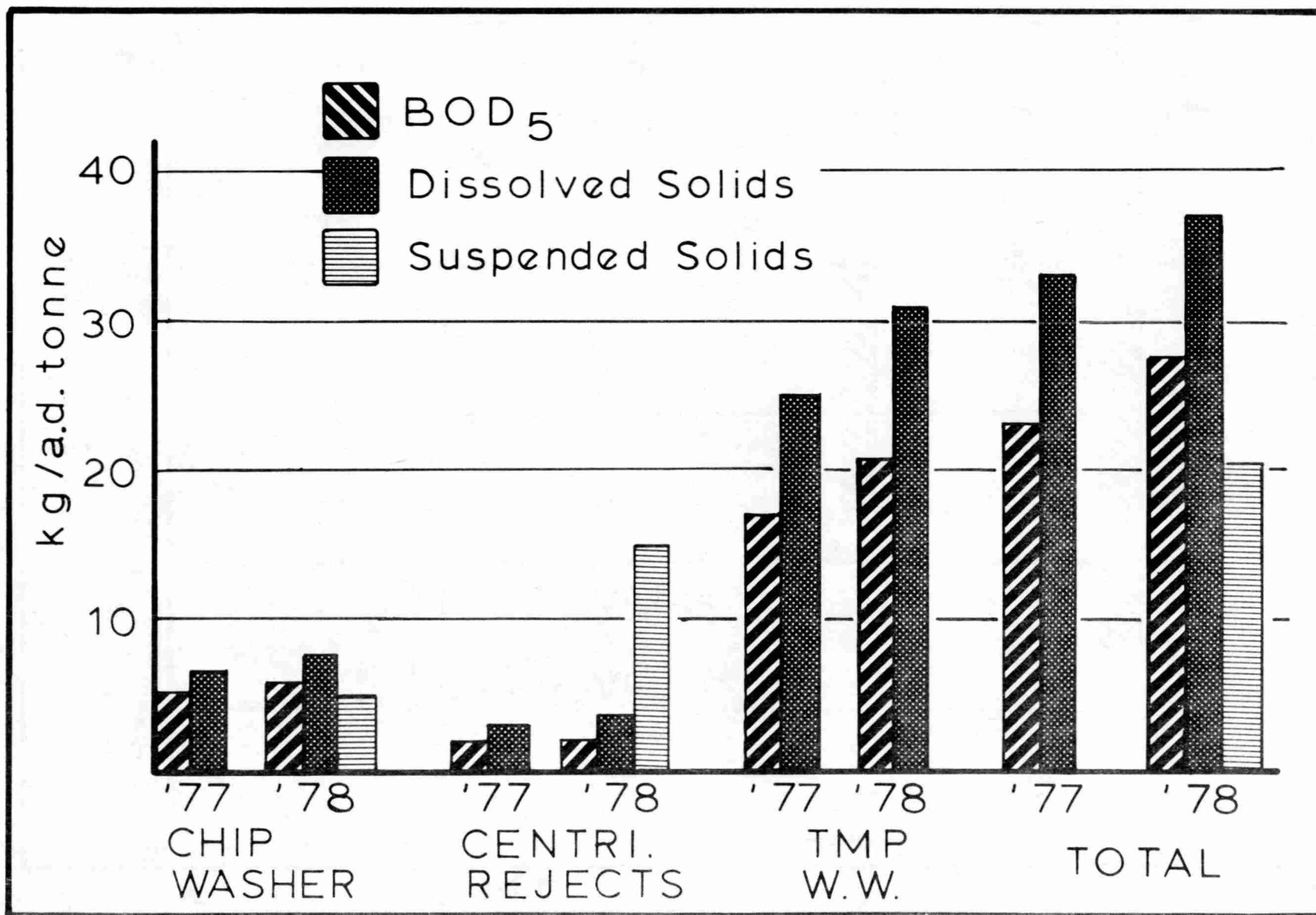


FIGURE 5  
EFFLUENT LOADINGS

Ontario



FIGURE 6

EFFLUENT LOADINGS



Ontario

(Organic Acids)

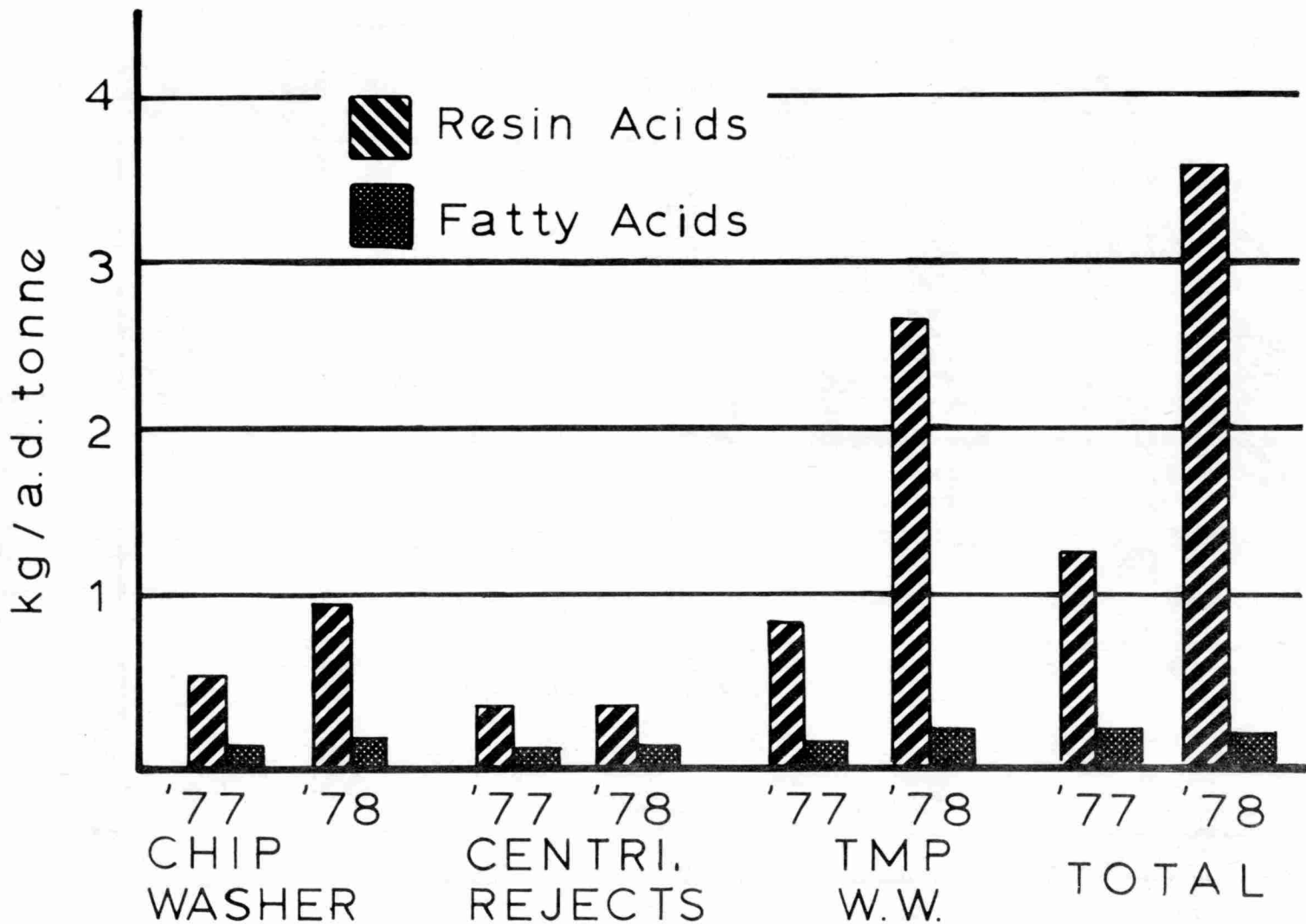




FIGURE 7

TREATABILITY STUDY

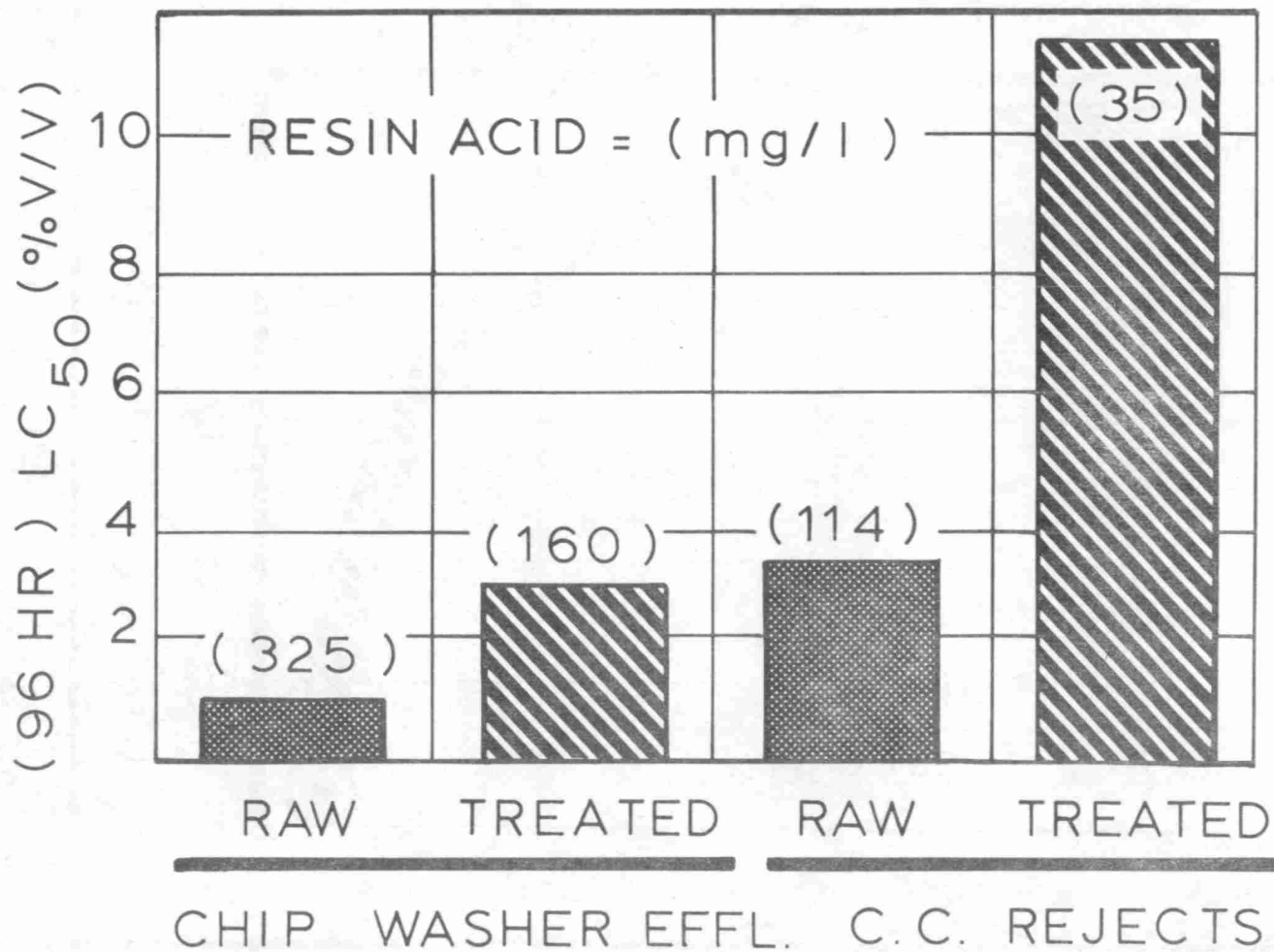
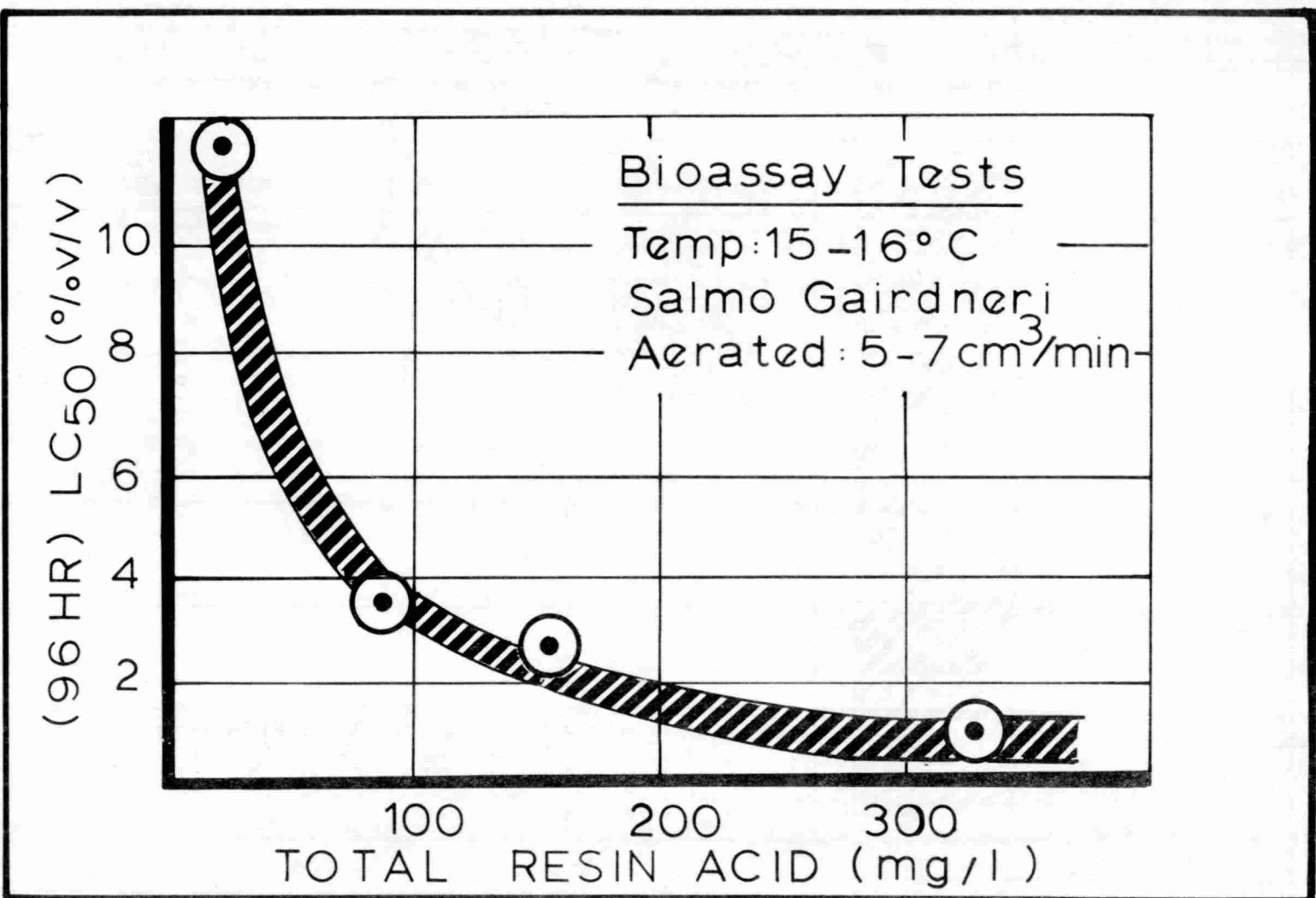


FIGURE 8

TOXICITY

VS.

CONCENTRATION



Ontario

TABLE 1: TMP mill water balance  
showing input/output accountability.

<u>WATER BALANCE</u> (litres/min)								
Date	Fresh Water	Steam	Chip Moist.	Total IN	TMP W.W.	TMP Sewer	Total OUT	Balance
July13	5334	8	95	5437	3936	1582	5518	101.3 %
" 14	5704	8	102	5814	4705	802	5507	94.7 %
" 15	5537	8	98	5643	4784	802	5586	99.0 %
" 16	6029	8	106	6143	5470	942	6412	104.4 %
					AVERAGE			99.9 %

TABLE 2: Showing effluent loadings of commonly measured parameters.

		BOD <sub>5</sub>		DISS. SOLIDS		SUSP. SOLIDS	
		1977	1978	1977	1978	1977	1978
+	CHIP WASHER EFFLUENT	4.0	5.5	6.3	6.7	—	5.6
	CENTRI. REJECTS	1.4	1.9	2.6	2.7	—	14.7
	TMP WHITEWATER	15.0	20.4	26.3	31.3	—	—
	sub.	20.4	27.8	35.2	40.7	—	20.3
-	INPUT HOT WATER	0.3	0.4	2.1	4.1	—	0.7
	NET DISCHARGE	20.1	27.4	33.1	36.6	—	19.6

(All loadings, kg/adt )

TABLE 3 : Organic Acids, generation rates

		<u>RESIN ACIDS</u>	<u>FATTY ACIDS</u>		
		<u>1977</u>	<u>1978</u>	<u>1977</u>	<u>1978</u>
+	CHIP WASHER EFFLUENT	0.52	0.87	0.05	0.17
	CENTRI. REJECTS	0.06	0.33	0.01	0.10
	TMP WHITEWATER	<u>0.75</u>	<u>2.85</u>	<u>0.01</u>	<u>0.10</u>
	sub.	<u>1.33</u>	<u>4.05</u>	<u>0.07</u>	<u>0.37</u>
-	INPUT HOT WATER	0.01	0.40	0.01	0.03
	NET DISCHARGE	<u>1.32</u>	<u>3.65</u>	<u>0.06</u>	<u>0.34</u>

( All loadings, kg/adt )

TABLE 4 : TMP mill effluents before  
and after treatment to reduce  
organic acid concentrations.

	CHIP WASHER		CENTRICLEANER REJECTS	
	<u>Before Treatment</u>	<u>After Treatment</u>	<u>Before Treatment</u>	<u>After Treatment</u>
	<u>( concentration; mg/l )</u>			
RESIN ACIDS	325	160	114	35
FATTY ACIDS	9.4	5.1	4.3	1.7

APPENDIX I

DETAILED DATA

- 1977 SURVEY

APPENDIX I - 1977 DATA

TABLE 1

TOTAL LOADINGS FROM PROCESS - 1977

B.O.D.<sub>5</sub> LOADING

TMP WITH NO BLEACH: (lb/Ton)

Chip Washing Effluent	8.0
Centricleaner Effluent	2.8
Stock Liquor to P.M.	30.1
	<hr/>
	40.9

Less raw water - 0.6

Net Loading = 40.3

TMP WITH BLEACH :

Chip Washing Effluent	8.0
Centricleaner Effluent	2.8
Stock Liquor to P.M.	33.0
	<hr/>
	43.8

Less raw water - 0.6

Net Loading = 43.2

DISSOLVED SOLIDS LOADING

TMP WITH NO BLEACH: (lb/Ton)

Chip Washer Effluent	12.5
Centricleaner Effluent	4.6
Stock Liquor to P.M.	52.6
	<hr/>
	69.7

Less raw water - 4.2

Net Loading = 65.5

TMP WITH BLEACH :

Chip Washer Effluent	12.5
Centricleaner Effluent	4.6
Stock Liquor to P.M.	57.6
	<hr/>
	74.7

Less raw water - 4.2

Net Loading = 70.5



APPENDIX I - 1977 DATA

TABLE 2

SUMMARY OF CHEMICAL PARAMETERS - 1977

	<u>B.O.D.5</u>				<u>Total Solids</u>			<u>Suspended Solids</u>			<u>Dissolved Solids</u>				<u>pH</u>	
	(mg/l)	(lb/d)	(lb/ADT)	(N)	(mg/l)	(lb/d)	(N)	(mg/l)	(lb/d)	(N)	(mg/l)	(lb/d)	(lb/ADT)	(N)		(N)
Process Warm Water	11	114	0.6	3	96	991	3	15	155	3	106	805	4.2	3	7.6	3
Chip Washer Effluent	1533	1528	8.0	3	1895	1888	3	1163	1163	3	2390	2381	12.5	3	5.2	3
Fourth Stage Rejects	995	539	2.8	4	11006	5946	4	9373	9373	4	1634	283	4.6	4	5.5	4
TMP Stock Liquor (No Brightener)	540	5711	30.1	1	1010	10682	1	-	-	-	945	9994	52.6	1	6.3	1
TMP Stock Liquor (With Brightener)	760	6277	33.0	1	1390	11480	1	-	-	-	1325	10944	57.6	1	4.7	1

T - Trace    N - Number of Samples    ND- Not Detectable    ADT - Air Dry Ton

# APPENDIX I - 1977 DATA

## TABLE 3

### SUMMARY OF ORGANIC ACID PARAMETERS - 1977

#### RESIN ACIDS

(all concentrations mg/l)	Process Warm Water	(N)	Chip Washer Effluent	(N)	Fourth Stage Rejects	(N)	TMP Stock (No Brightener)	(N)	TMP Stock (+ Brightener)	(N)
Pimaric	T	8	1.14	6	0.5	8	0.42	2	0.37	1
Sandaracopimaric	T	8	1.42	5	0.92	6	-		0.49	1
Levopimaric	T	8	8.47	4	6.76	7	1.89	2	1.67	1
Isopimaric	T	8	4.25	8	1.26	8	0.82	2	0.87	1
Abietic	T	8	82.9	8	34.96	8	24.47	2	25.52	1
Dehydroabietic	T	8	11.2	6	2.12	6	T	2	6.69	1
Total	T-0.13	8	99.2	8	46.03	8	27.6	2	35.61	1
Loading (lb/ADT)	-		0.5		0.11		1.5		1.5	

#### FATTY ACIDS

(all concentrations mg/l)										
Capric	T-0.06	8	0.81	5	T-0.88	8	ND	2	ND	
Lauric	T-0.67	8	2.27	6	0.6	8	0.24	2	0.19	
Myristic	0.03	8	0.41	8	T-0.10	8	ND	2	ND	
Palmitic	T	7	0.75	3	0.86	3	0.26	2	0.24	
Stearic	T	7	ND	4	ND-0.30	7	ND	2	ND	
Linoleic	0.04	8	3.58	8	1.86	8	0.92	2	1.37	
Linoleic	T	8	ND	4	0.30	8	ND	2	ND	
Arachidic	T	8	4.96	8	1.34	8	ND	2	ND	
Oleic	T	8	1.20	3	0.54	7	0.44	2	0.56	
Total	T-0.36	8	9.35	8	4.34	8	0.86	2	2.36	
Loading (lb/ADT)	-		0.10		0.01		0.10		0.10	

T - Trace    N - Number of Samples

ND - Not Detectable

ADT - Air Dry Ton

APPENDIX I - 1977 DATA

TABLE 4

SUMMARY OF TOXICITY RESULTS - 1977

	<u>Process Warm Water</u>	<u>Chip Washer Effluent</u>	<u>Fourth Stage Rejects</u>	<u>TMP Stock No Brightener</u>	<u>TMP Stock With Brightener</u>
Toxicity (96 hr. LC <sub>50</sub> )	Non-lethal in 100%(v/v)	2%	2%	2%	2%
95% Confidence Limit	-	-	-	4.13-0.92	4.05-0.15
Median Survival Time (Hrs)					
2%	-	6.4	12	72	
5%	-	4.5	17	40% mortality in 96 hrs.	10% mortality in 96 hrs.
10%	-	1.7	-	15	8.5
20%	-	0.9	2.5	5.2	5.2
50%	-	0.9	1.7	2.2	2.9

TABLE 5

Water Quality Parameters for L. Panache water  
used as diluent for bioassays.

<u>Parameter</u>	<u>Concentration</u>
pH	6.9
Ca	7.4 ug/l
Na	1.8 ug/l
K	0.8 ug/l
Sulphate	21. mg/l
Hardness	24. mg/l as CaCO <sub>3</sub>
Alkalinity	8.0 mg/l as CaCO <sub>3</sub>
Conductivity	74 umhos/cm <sup>2</sup> @ 15°C
Chloride	2.0 mg/l
Total Chlorine Residue	None

APPENDIX I- 1977 DATA

TABLE 6

BASIC CHEMICAL RESULTS - 1977

PROCESS WARM WATER

<u>Lab. No.</u>	<u>B.O.D.5</u>		<u>Total Solids</u>		<u>Susp. Solids</u>		<u>Diss. Solids</u>		pH	<u>Cond.</u> uMHo/cm <sup>2</sup>	<u>Flow</u> (USGM) (2)
	(mg/l)	(lb/D)	(mg/l)	(lb/D)	(mg/l)	(lb/D)	(mg/l)	(lb/D)			
T24-81	12	124	125	1291	15	155	109	1125	7.6	140	860
T24-86	12	124	120	1239	15	155	104	1074	7.7	139	860
T24-153	10	103	45	45	15	155	20 (1)	206	7.6	142	860
<hr/>											
Average	11	114	96	991	15	155	106	805		140	860

B.O.D.5/D.S. Ratio: Company average = 1.4  
O.M.O.E. = 1.1

- (1) Data appears uncharacteristic. Omitted from average results.
- (2) Flow rate information provided by company.

APPENDIX I - 1977 DATA

TABLE 7

BASIC CHEMICAL RESULTS - 1977 DATA

CHIP WASHER EFFLUENT

<u>Lab. No.</u>	<u>B.O.D.<sub>5</sub></u>		<u>Total Solids</u>		<u>Susp. Solids</u>		<u>Diss. Solids</u>		pH	<u>Cond.</u> uMHO/cm <sup>2</sup>	<u>Flow</u> (USGM) (2)
	(mg/l)	(lb/D)	(mg/l)	(lb/D)	(mg/l)	(lb/D)	(mg/l)	(lb/D)			
T24-77	1,600	1,594	3,410	3,398	1,075	1,071	2,335	2,326	5.2	190	83
T24-82	1,400	1,395	3,410	3,398	1,065	1,061	2,345	2,337	5.2	193	83
T24-146	1,600	1,594	3,840	3,826	1,350	1,345	2,490	2,481	5.1	215	83
T24-153	660(1)	658(1)	1,895(1)	1,888(1)	265(1)	264(1)	1,630(1)	1,624(1)	5.5(1)	146(1)	83
Average	1,533(2)	1,528	3,553	3,540	1,163	1,159	2,390	2,381		199	83

B.O.D.<sub>5</sub>/D.S. Ratio: Company Average = .96

O.M.O.E. = .64

(1) Refiners down - Data omitted from averages

(2) B.O.D.<sub>5</sub> data is lower than company 'historical' information for this effluent. Company data: B.O.D.<sub>5</sub> 3325 ± 764 mg/l

(3) Flow rate information provided by company.

APPENDIX I - 1977 DATA

TABLE 8

BASIC CHEMICAL RESULTS - 1977

FOURTH STAGE CLEANER REJECTS

<u>Lab.No.</u>	<u>B.O.D.5</u>		<u>Total Solids</u>		<u>Susp. Solids</u>		<u>Diss. Solids</u>		pH	<u>Cond.</u> uMHO/cm <sup>2</sup>	<u>Flow</u> (USGM) (1)
	(mg/l)	(lb/D)	(mg/l)	(lb/D)	(mg/l)	(lb/D)	(mg/l)	(lb/D)			
T24-79	1,250	675	12,210	6,596	10,585	5,718	1,625	878	5.5	139	45
T24-84	950	513	12,060	6,515	10,390	5,670	1,670	902	5.5	144	45
T24-150	900	486	8,750	4,727	6,965	1,785	1,785	964	5.3	148	45
T24-157	880	475	11,005	5,945	9,550	5,159	1,455	786	5.5	133	45
Average	995	538	11,006	5,946	9,373	5,064	1,634	883		141	45

B.O.D.5/D.S. Ratio: Company Average = .74

O.M.O.E. = .61

(1) Flow rate information provided by company.

APPENDIX I - 1977 DATA

TABLE 9

BASIC CHEMICAL RESULTS - 1977

TMP STOCK (No Brightener Added)

<u>Lab.No.</u>	<u>B.O.D.5</u> (mg/l) (lb/D)		<u>Total Solids</u> (mg/l) (lb/D)		<u>Diss. Solids</u> (mg/l) (lb/D)		<u>C. O. D.</u> (mg/l) (lb/D)		pH	<u>Cond.</u> uMHO/cm <sup>2</sup>	<u>Flow</u> (USGM)
T24-159	540	5,711	1010	10,682	945	9,994	1140	12,057	6.3	215	881(1)

B.O.D.5/D.S. Ratio: Company average: .65  
O.M.O.E. : .57

TMP STOCK (With Brightener Added)

T24-160	760	6,277	1390	11,480	1,325	10,944	1650	13,628	4.7	300	688(1)
---------	-----	-------	------	--------	-------	--------	------	--------	-----	-----	--------

B.O.D.5/D.S. Ratio: Company average: .66  
O.M.O.E. : .57

- (1) Flow rate of stock from high density storage tank calculated from production rate and stock consistency data. For details see Table 4. Note; Bleached and unbleached stock flows can vary since pumping to paper mill is dependent upon demand and not solely on TMP production rate.
- (2) Suspended Solids data appears for TMP stock that was 'rough' filtered for analysis of chemical parameters.



APPENDIX I - 1977 DATA

TABLE 10

CALCULATION OF TMP STOCK FLOW - 1977

Basis: Refiner rate : 200 T/D  
Yield : 95%  
Net Production : 190 ADT/D (Air Dry)  
: 171 BDT/D (Bone Dry)  
Stock Consistency: 3.12% (No Bleach Added)  
: 3.96% (With Bleach Added)

TMP STOCK (No Bleach Added)

Total Flow to Paper mill:

$$.9 \times 190 \times \frac{100}{3.12} = 5480.8 \text{ BDT/D}$$

$$5481 - 190 = 5291 \text{ tons/D}$$

$$\frac{5291 \times 2000}{10^6} = 10.582 \text{ mill. \#/D}$$

$$\frac{10.582 \times 10^6}{8.337 \times 1440} = 881 \text{ U. S. Gallons/min.}$$

TMP STOCK (No Bleach Added)

Total Flow to Paper mill:

$$.9 \times 190 \times \frac{100}{3.96} = 4318.2 \text{ BDT/D}$$

$$4318 - 190 = 4128 \text{ tons/D}$$

$$\frac{4128 \times 2000}{10^6} = 8.256 \text{ mill. \#/D}$$

$$\frac{8.256 \times 10^6}{8.337 \times 1440} = 688 \text{ U.S. Gallons/Min.}$$

APPENDIX II

DETERMINATION OF FLOW BY SALT  
DILUTION TECHNIQUE

## APPENDIX II

### DETERMINATION OF FLOW BY SALT DILUTION TECHNIQUE

In a flowing system the injection of a tracer at a constant rate causes a resultant final concentration of tracer to occur. As can be seen by the following equation, the resultant tracer concentration is directly proportional to the flow in the system, since:

$$Q = q \frac{C^*}{c}$$

Where: Q= flow (l/min)  
q= tracer injection rate (l/min)  
C= initial tracer concentration (gm/l)  
c= resultant tracer concentration (gm/l)

\* It is important to realize that the injection flow rate must be negligible when compared to the flow being measured (1)

Sodium chloride solutions were used in the TMP mill to determine the flows of the incoming fresh water and the outgoing pulp stock being pumped to the adjacent news-print mill.

---

(1) Adapted from: Fluorometric Facts; Flow Measurement  
Turner Designs Incorporated, California 94043

Highly concentrated solutions of NaCl were prepared and analysed. Samples of the stream to be measured were acquired and background sodium concentrations determined. A Perkin-Elmer Model 111 photometer was set up to analyse samples at the mill.

Based upon the observed background concentrations noted an injection rate was determined. An estimated flow in the stream to be calibrated was used to calculate required injection rates such that the resultant Na<sup>+</sup> concentration would be 15-25 times higher than background.

Injection of salt solutions were accomplished by using a pressurized metering pump which charged tracer directly into the stream being measured. The beginning of the injection was co-ordinated to correspond to the start of sampling, and the recording of mill flow measuring equipment readings. Samples were collected over a sufficient length of time, and analysed while the test progressed in order to observe fluctuations in resultant salt concentrations. Since instability in salt concentration would have indicated improper mixing, a positive indication of a correct injection location was provided.

Appendix II (i) and II (ii) describe these calibration tests in detail.

Data from the test was then used to calculate the flow of material in the stream during the study. These observed flows were used to calculate a correction factor for mill flow measuring equipment. Several tests were performed at each source to test reproducibility of the technique.

APPENDIX II(i)

CALIBRATION OF TMP MILL

FRESH WATER FLOW

APPENDIX II(i)

CALIBRATION OF FRESH WATER FLOW

As previously discussed salt solution was injected at a constant rate into the TMP fresh water strainer. This device is located after the fresh water and is a sieve device which rough filters incoming water. Figure 1 illustrates the experimental set-up used in this test.

The first test performed was broken into two parts due to an experimental problem. Test details and observed Na<sup>+</sup> concentrations are listed in Table 1. Note that two sampling locations are listed. Two sites were sampled in order to test for complete mixing of tracer.

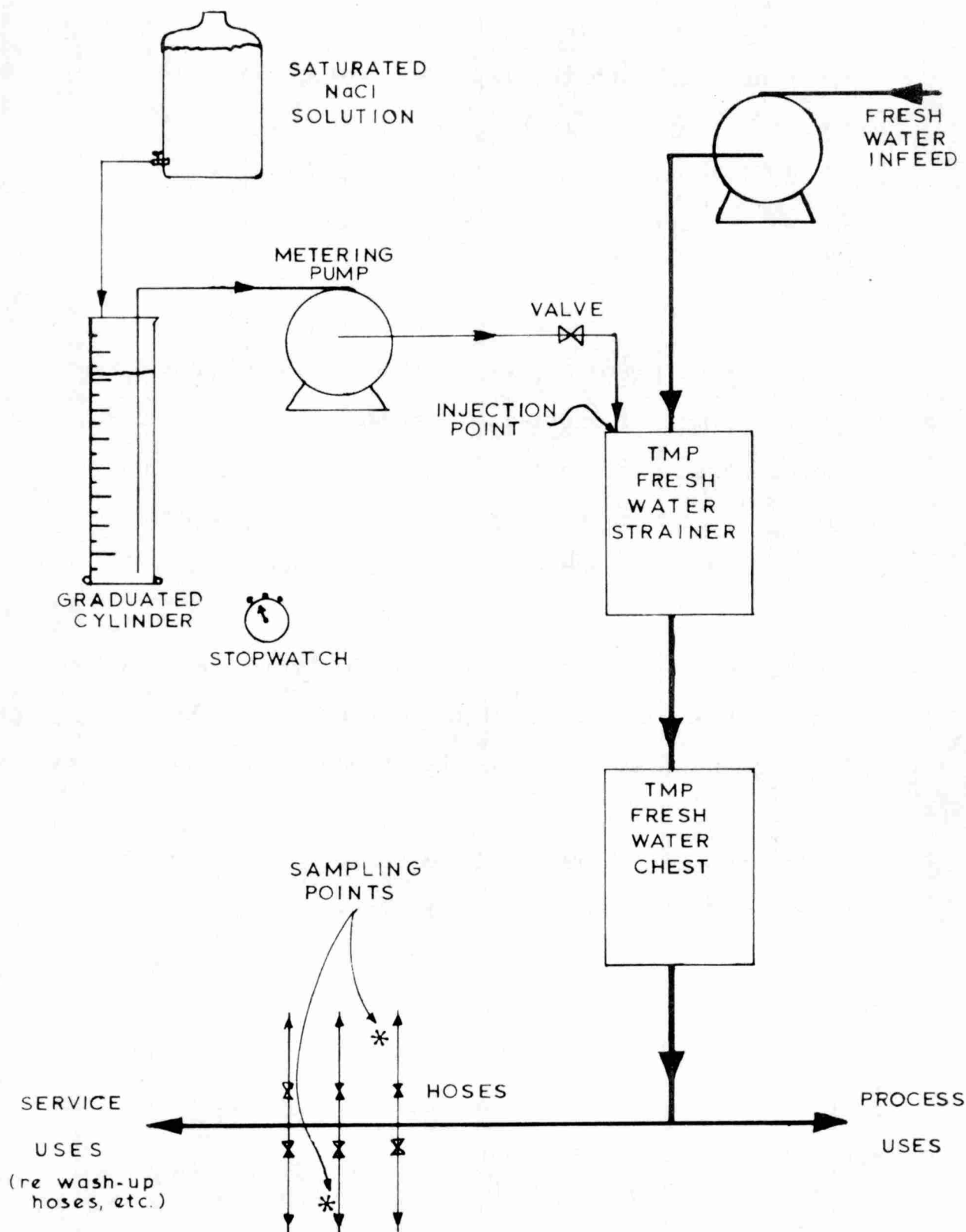
A second test was performed on the following day from the first calibration. Table 2 indicates the stability of injection and that complete mixing had been accomplished.

Calculations to determine the corresponding flows during both tests follow Table 1 and Table 2.

The correction factors arrived at for the three tests were: 1.49, 1.43, and 1.25 respectively. The average correction factor was 1.39 or 46.4 U.S. gallons per integrator count. ( $.175 \text{ m}^3/\text{count}$ ).

# FIGURE 1

## TMP FRESH WATER CALIBRATION





APPENDIX II (i)

TABLE 1

FRESH WATER FLOW CALIBRATION

(Test A- July 13, 1978)

Time (min)	Resultant Na <sup>+</sup> Conc.			Tracer Injection			Mill Instruments (count)
	Site A (mg/l)	Site B (mg/l)	Aver. (mg/l)	Volume (l)	Sol'n A (ml/min)	Sol'n B (ml/min)	
0	-	-	-	12.8	-	-	700921
1	1	1	1	11.8	1000	-	
2	1	1	1	10.8	1000	-	
3	1	1	1	10.0	800	-	
4	1	1	1	9.0	1000	-	
5	1	1	1	8.0	1000	-	
6	1	1	1	-	960	-	
7	1	1	1	7.2	-	-	
8	1	3	2	6.3	900	-	
9	1	8	4.5	6.2	-	-	
10	7	11	9.0	5.3	900	-	
11	11	14	12.5	4.4	900	-	
12	14	15	14.5	3.4	1000	-	
13	15	15	15.0	2.4	1000	-	
14	15	15	15.0	lost suction on pump			
15	15	15	15.0	re-equilibrating injection rate			
16	15	15	15.0				
17	15	9	12.0	Aver.=13.5	-	-	
18	11	4	7.5		-	-	
19	4	8	6.0	Aver.=14.5	11.1	1000	
20	5	12	13.0		10.2	900	
21	12	14	15.0	Aver.=14.5	9.2	1000	
22	15	15	15.0		8.2	1000	
23	15	15	15.0	Aver.=14.5	-	-	
24	15	15	15.0		6.8	1000	
29	Average				951	950	701823

Integrator Difference

902\*\*

\* Solution A = 74.00 gm/l; Solution B = 83.00 gm/l as Na<sup>+</sup>

\*\* Counts per minute =  $\frac{902}{29}$  counts = 31.1 min.

APPENDIX II (i)

TABLE 2

FRESH WATER FLOW CALIBRATION

(Test B - July 14, 1978)

Time (min)	Resultant Na <sup>+</sup> Conc.			Tracer Injection		Mill Instruments (count)
	Site A (mg/l)	Site B (mg/l)	Aver. (mg/l)	Volume (l)	Sol'n C (ml/min)	
0	-	-	-	17.5	-	726936
1	1	1	1	-	-	
2	1	1	1	14.75	1375	
3	1	1	1	13.40	1350	
4	1	1	1	11.95	1450	
5	1	2	1.5	10.65	1300	
6	1	19	10.0	9.30	1350	
7	1	20	18.5	8.00	1300	
8	16	21	22.0	6.65	1350	
9	22	22	22.5	5.30	1350	
10	21	24	22.0	3.90	1400	
11	21	23	23.0	2.50	1400	
12	22	24	23.0	-		
13	22	24	23.0	16.40		
14	25	24	24.5	-		
15	23	24	23.5	13.62	1390	
16	23	24	23.5	-		
17	23	27	25.0	10.90	1370	
18	24	23	23.5	-		
19	22	23	22.5	-		
20	23	24	23.5	6.70	1400	
21	24	24	24	-		
22	24	25	24.5	3.95	1380	
23	24	25	24.5	-		
24	24	23	23.5	1.20	1370	
25	24	25	24.5			
26	26	27	26.5			
27	26	-	-			
28	5	3	4			
30	1	1	1			
32	1	1	1			
34	1	1	1			
36	1	1	1			
			Average		1370	728051

Instrument Counts

1115\*\*

\* Solution C = 80.00 gm/l Na<sup>+</sup>

\*\* Counts per minute =  $\frac{1115}{36}$  counts = 31.0

APPENDIX II (i)

CALCULATIONS OF FLOW

Fresh Water Flow Calibration

Test #1 - Part (A)      July 13, 1978

- Time: t = 0, 12 minutes

Basis: Tracer injection flow = 951 ml/min

Tracer concentration = 74.00 g/l as Na<sup>+</sup>

Resultant concentration = 13.5 mg/l as Na<sup>+</sup> (average)

Background concentration = 1.0 mg/l as Na<sup>+</sup>

Observed

$$\text{Flow:} = .951 \frac{\text{l}}{\text{min}} \times \frac{74000 \text{ mg/l}}{(13.5 - 1) \text{ mg/l}}$$

$$= 5630 \frac{\text{l}}{\text{min}} \times \frac{1 \text{ US gallon}}{3.785 \text{ l}}$$

$$= 1487 \text{ US gallons/minute (8094 m}^3\text{/d)}$$

Indicated

$$\text{Flow:} = 31.1 \frac{\text{counts}}{\text{min.}} \times \frac{100 \text{ US gallons}}{3 \text{ counts}}$$

$$= 1037 \text{ US gallons/minute (5645 m}^3\text{/d)}$$

Calibration

$$\text{Factor:} = \frac{1487 \text{ USGM (observed)}}{1037 \text{ USGM (indicated)}} = 1.434$$

$$\text{or } 1 \text{ count} = \frac{100}{3} \times 1.434 = 47.8 \text{ USGM (260 m}^3\text{/d)}$$

APPENDIX II (i)

CALCULATIONS OF FLOW

Fresh Water Flow Calibration

Test #1 - Part (B) July 13, 1978

- Time: t = 19, 29 minutes

Basis: Tracer Injection Flow = 950 ml/min

Tracer Concentration = 83.00 g/l as Na<sup>+</sup>

Resultant Concentration = 14.5 mg/l as Na<sup>+</sup> (average)

Background Concentration = 1.0 mg/l as Na<sup>+</sup>

Observed

$$\text{Flow:} = \frac{950 \text{ l}}{\text{min}} \times \frac{83000 \text{ mg/l Na}^+}{(14.5 - 1) \text{ mg/l Na}^+}$$

$$= \frac{5841 \text{ l}}{\text{min}} \times \frac{1 \text{ US gallon}}{3.785 \text{ l}}$$

$$= 1543 \text{ US gallons/min (8410 m}^3\text{/d)}$$

Indicated

$$\text{Flow:} = 31.1 \times \frac{100}{3} = 1037 \text{ US gallons/min (5645 m}^3\text{/d)}$$

Calibration

$$\text{Factor:} = \frac{1543}{1037} \frac{\text{USGM}(\text{observed})}{\text{USGM}(\text{indicated})} = 1.488$$

$$\text{or 1 count} = 100 \times 1.488 = 49.6 \text{ USGM (270 m}^3\text{/d)}$$

APPENDIX II (i)

CALCULATION OF FLOW

Fresh Water Flow Calculation

Test #2      July 14, 1978

Time: t = 0 , 36 minutes

Basis:            Tracer Injection Flow      = 1370 m/min  
                    Tracer Concentration      = 80.00 g/l  
                    Resultant Concentration   = 23.4 mg/l as Na<sup>+</sup> (average)  
                    Background Concentration = 1 mg/l as Na<sup>+</sup>

Observed  
Flow:      =  $1.370 \frac{\text{l}}{\text{min}} \times \frac{8000 \text{ mg/l as Na}^+}{(23.4 - 1) \text{ mg/l as Na}^+}$   
                 =  $4893 \frac{\text{l}}{\text{min}} \times \frac{1 \text{ US gallon}}{3.785 \text{ l}}$   
                 = 1293 US gallons/min (7038 m<sup>3</sup>/d)

Indicated  
Flow:      =  $31.0 \times \frac{100}{3} = 1033 \text{ US gallons/min (5623 m}^3\text{/d)}$

Calibration  
Factor:   =  $\frac{1293 \text{ USGM (observed)}}{1033 \text{ USGM (indicated)}} = 1.252$

or 1 count =  $\frac{100}{3} \times 1.252 = 41.7 \text{ USGM (227 m}^3\text{/d)}$

APPENDIX II(ii)

DETERMINATION OF TMP MILL

STOCK FLOW TO PAPER MILL

APPENDIX II (ii)

DETERMINATION OF STOCK FLOW TO PAPER MILL

The salt dilution technique previously described was employed in calibrating mill equipment which indicated flow of pulp to the paper mill. Salt solution was injected at the suction side of a 'booster' pump moving pulp from the TMP unit.

The calibration test for pulp stock was done in two sections. Each period represented a different injection rate. Figure 1 illustrates the experimental set-up used for this study.

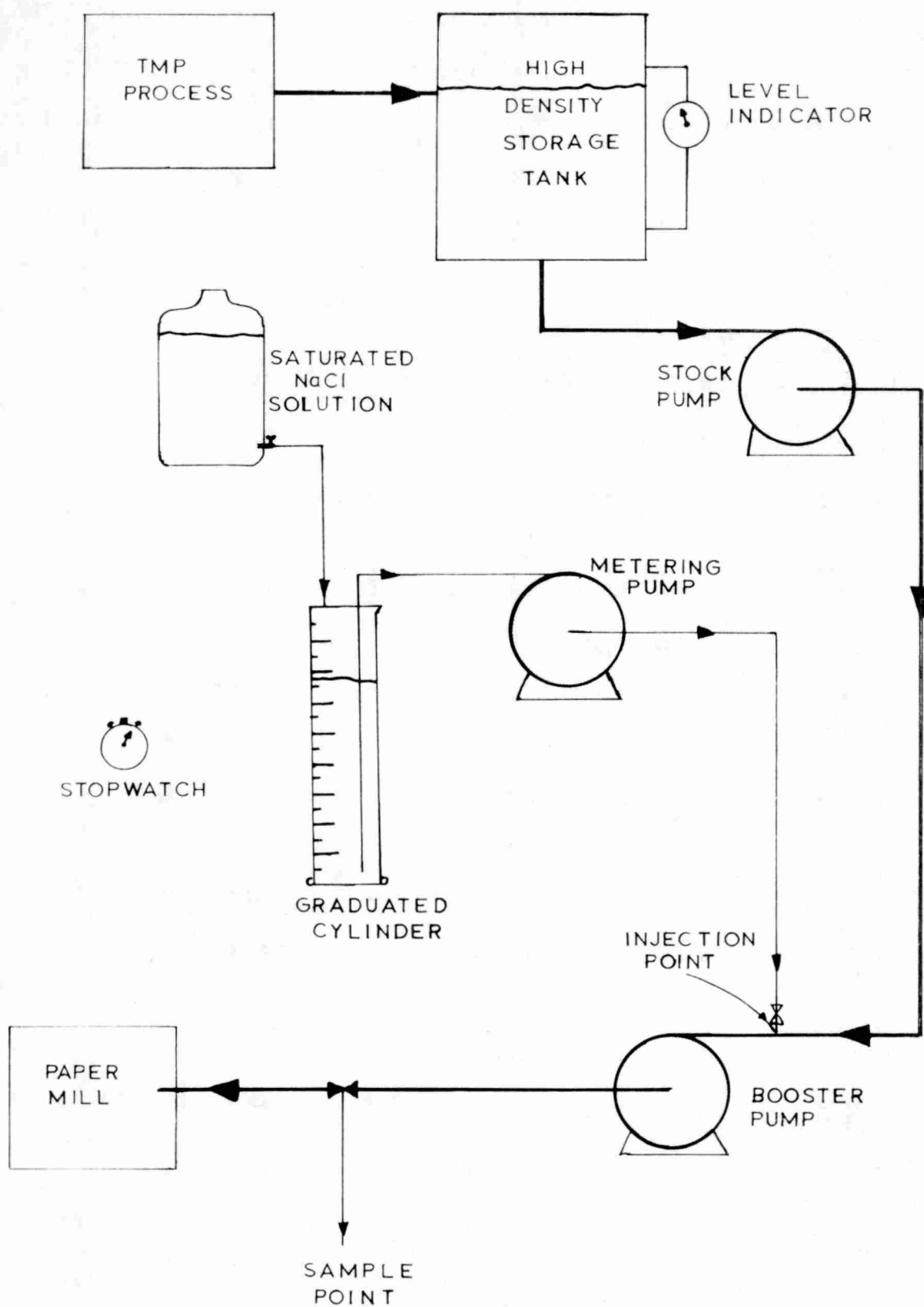
Table 1 indicates the stability of the tracer injection for the two portions of the test - Part (A)  $t = 0, 14$  minutes; and Part (B) 17, 36 minutes. It should be noted that the injection rates were kept constant by adjusting the metering pump as required.

Calculations to determine the flow indicated by the observed tracer concentrations follow Table 1.

The correction factors determined by the test were: 1.234 and 1.346 respectively. The average correction factor was 1.291 or 129 US gallons per count ( $.488 \text{ m}^3/\text{count}$ ).

# FIGURE 1

## TMP STOCK FLOW CALIBRATION





APPENDIX II(ii)

TABLE 1

STOCK FLOW CALIBRATION TESTS

Time (min)	Resultant Na <sup>+</sup> Conc. (mg/l)	Tracer Injection		Mill Instruments (counts)
		Volume (l)	Sol'n D* Sol'n E* (ml/min) (ml/min)	
0	-	17.20	-	139890
1	3	15.80	1400	
2	21	14.50	1300	
3	22	13.20	1300	
4	21	11.85	1350	
5	20	10.55	1300	
6	21	9.30	1250	
7	21	8.15	1150	
8	20	7.05	1100	
9	19	5.90	1150	
10	-	4.90	1000	
11	18	3.90	1000	
12	17	2.90	1000	
13	17	1.90	1000	
14	16	0.90	1000	
15	16	17.50	-	
16	15	16.80	700	
17	14	-	780	
18	13	15.25	750	
19	14	14.50	700	
20	12	13.80	700	
21	-	13.20	600	
22	13	12.50	700	
23	14	11.80	700	
24	-	11.05	750	
25	13	10.40	650	
26	-	9.70	700	
27	12	9.00	700	
28	12	8.40	600	
29	-	7.60	800	
30	12	7.00	600	
31	-	6.35	650	
32	12	5.55	800	
33	13	4.85	700	
34	14	4.10	750	
35	-	3.35	750	
36	15	2.50	850	140303
37	-	1.70		
38.	1			
38.5	1			
39.5	1			
40.5	1			
Average		1160	720	413**
Instrument Counts				

\* Solution D- 74.00 g/l as Na<sup>+</sup>; Solution E - 80,000 g/l Na<sup>+</sup>

\*\* Counts per minute =  $\frac{413}{37} = 11.2$

APPENDIX II (ii)

TABLE

CALCULATION OF FLOW

STOCK FLOW TO PAPER MILL

Part A

- Time: t=2, 14 minutes July 15, 1978

Basis:

Tracer injection flow: 1160 ml/min.

Tracer Concentration: 74.00 g/l as Na<sup>+</sup>

Resultant Concentration: 19.4 mg/l as Na<sup>+</sup>  
(average)

Background Concentration: 3 ml as Na<sup>+</sup>  
(average)

Observed Flow:

$$\begin{aligned} & 1.160 \frac{\text{l}}{\text{min}} \times \frac{7400 \text{ mg/l}}{(19.4 - 3) \text{ mg/l}} \\ &= 5234 \frac{\text{l}}{\text{min}} \times \frac{1}{3.785} \frac{\text{US gallon}}{\text{l}} \\ &= 1383 \text{ US gallons/min (7528 m}^3\text{/d)} \end{aligned}$$

Indicated Flow:

$$\begin{aligned} & \frac{413}{37} \times 100 \frac{\text{US gal}}{\text{count}} \\ &= 1120 \text{ US gallons/min (6096 m}^3\text{/d)} \end{aligned}$$

Calibration Factor:

$$\begin{aligned} & \frac{1383 \text{ USGM (observed)}}{1120 \text{ USGM (indicated)}} = 1.234 \\ & \text{Or } 1 \text{ count} = 100 \times 1.234 = 123.4 \text{ USGM} \\ & \quad \quad \quad (.466 \text{ m}^3\text{/count}) \end{aligned}$$

APPENDIX II(ii)

CALCULATION OF FLOW

STOCK FLOW TO PAPER MILL

Part B

Time: t = 17, 36 minutes July 15, 1978

Basis:

Tracer injection flow: 720 m/min

Tracer Concentration: 80.00 g/l as Na<sup>+</sup>

Resultant Concentration: 13.1 mg/l as Na<sup>+</sup>  
(average)

Background Concentration: 3.0 mg/l as Na<sup>+</sup>

Observed Flow:

$$\begin{aligned} & 0.720 \frac{1}{\text{min}} \times \frac{80000 \text{ mg/l}}{(13.1 - 3) \text{ mg/l}} \\ &= 5703 \frac{1}{\text{min}} \times \frac{1}{3.785} \text{ US gallon} \\ &= 1507 \text{ US gallons/min. (8203 m}^3\text{/d)} \end{aligned}$$

Indicated Flow:

as per Part A = 1120 USGM  
(6096 m<sup>3</sup>/d)

Calibration Factor:  $\frac{1507}{1120} \frac{\text{USGM (observed)}}{\text{USGM (indicated)}} = 1.346$

or 1 count = 100 x 1.346 = 134.6 USGM  
(.509 m<sup>3</sup>/count)

APPENDIX III

THE ANALYSIS OF RESIN AND FATTY ACIDS IN  
THERMOMECHANICAL PULP MILL EFFLUENTS

Organic Trace Contaminants Section  
Laboratory Services Branch  
Ontario Ministry of the Environment

by:

E. G. Adamek and L. Au - April 1978

## ANALYTICAL PROCEDURES

### A. Principle of Analytical Method

The weakly acidified (pH3) aqueous samples from TMP process effluents are extracted with diethyl ether. The ether extract containing the resin and fatty acids is separated, dried and concentrated to small volume. By reaction with diazomethane, these organic acids are converted to the corresponding methyl esters. By gas chromatography with the use of a flame ionization detector and with temperature programming, the methyl esters are separated on a column of SP216 on Supelcoport. By relating the peak heights of the esters on the gas chromatogram to those of known standards, quantitative estimation of the organic acid components in the original samples is possible even at the ppb level.

### B. Range and Sensitivity

The lower detection limit for the measurement of resin acids is 0.05 mg/litre and for fatty acids 0.02 mg/litre. There are no upper limit problems when measuring concentrations of these acids in aqueous process effluents.

### C. Interferences and Limitations

Extraction efficiencies for the resin and fatty acids were found in recovery experiments with standard solutions to be between 88% and 95%. In this preliminary work, the analytical results are reported as obtained, without adjustment to these minor losses.

In the gas chromatographic analysis, certain phenolic compounds may elute at similar retention time as the fatty acid esters. In the samples investigated, these phenolic compounds appeared to be present at much lower concentrations so that interferences with the fatty acid analyses was regarded as insignificant.

It should be noted that in this exploratory work, identification of the fatty and resin acids was by gas chromatographic retention time only and, therefore, should be regarded as tentative.

#### D. PROCEDURE

##### 1. Sample Pretreatment and Extraction

The aqueous samples of about 500 ml, as taken at the sampling points, are routinely adjusted to pH3 with concentrated hydrochloric acid. This treatment serves to preserve the samples from bacterial degradation and to ensure uniformity in the subsequent ether extraction of the organic acids.

The extraction procedure involves the following steps:

- a. Measure volume of sample (in ml) in a graduate cylinder and transfer the acidified sample into a 1000 ml separatory funnel.
- b. Add 100 ml of glass-distilled diethyl ether and shake 2 minutes. Then allow mixture to settle.
- c. Drain lower, aqueous portion into the original sample bottle. Then collect top layer of mixture in a 500 ml Erlenmeyer flask.
- d. Repeat the extraction procedure with another 50 ml portion of glass-distilled diethyl ether.
- e. Rinse sample bottle with 30 ml of diethyl ether and pour into the separatory funnel.
- f. Drain lower layer of mixture and discard. Collect top layer in the same Erlenmeyer flask.
- g. Rinse walls of separatory funnel twice with 20 ml portions of diethyl ether and transfer to the Erlenmeyer flask.
- h. Add approximately 8 gm of anhydrous magnesium sulphate to the ether extract in the Erlenmeyer flask and shake intermittently for about 10 minutes to dry the extract.
- i. Pass the ether extract through a glass fibre filter into a 300 ml round bottom flask.
- j. Rinse walls of Erlenmeyer flask twice with portions of anhydrous diethyl ether and collect in the round bottom flask.

- k. Concentrate extract in the round bottom flask to about 1 ml volume by using a rotary evaporator with a water-bath at room temperature. Stopper the flask and retain the 1 ml concentrate for the methylation procedure (see 2 b).

## 2. Esterification with Diazomethane

### a. Preparation of Diazomethane

CAUTION: Diazomethane is very toxic and may explode on heating or when concentrated from solutions. Use a fume hood, safety shield and safety glasses. Use "Teflon" sleeves in ground glass joints of glass apparatus.

Since diazomethane is not commercially available, it is prepared in the laboratory from intermediate compounds, such as p-tolylsulphonylmethylnitrosamide.

Into a 250 ml long-necked Claisen distilling flask, provided with a dropping funnel and an efficient downward condenser, place a solution of 12 g of potassium hydroxide in 20 ml water, followed by 70 ml of carbitol (diethyleneglycol monoethyl ether) and 20 ml of diethyl ether. Connect the condenser to a 100 ml round-bottom flask and a 100 ml conical flask, in series, containing 20 and 45 ml of diethyl ether respectively. While these flasks are cooled in an ice-salt bath, the mixture in the Claisen distilling flask is heated on a water-bath at about 70°C with constant stirring by means of a magnetic bar and stirrer.

As soon as the ether commences to distil, add a solution of 45 gm p-tolylsulphonylmethylnitrosamide in 250 ml of diethyl ether through the dropping funnel and distil until the distillate is colourless. The ethereal solution in the round-bottom flask contains about 6 gm of diazomethane. Replace the distilling flask with the round-bottom flask containing the diazomethane solution, and re-distil the diazomethane. The resulting ethereal solution of diazomethane can be stored in the freezer (at about - 28°C) if not immediately used.

b. Methylation with Diazomethane

- i. Add 1.5 ml of the diazomethane solution slowly to the round-bottom flask containing the 1 ml concentrate of the ether extract of the sample (see 1K) while being cooled in an ice bath. During this operation, the initial gas evolution ceases and the reaction mixture assumes a pale yellow colour.
- ii. Transfer the content of the flask to a 20 ml graduated conical glass tube.
- iii. Rinse the walls of the flask twice with several ml of diethyl ether and add to the conical glass tube.
- iv. Concentrate the content in the conical glass tube to exactly 1.0 ml volume by blowing down in a stream of nitrogen in a water-bath at room temperature.
- v. Use 5 ul aliquots for injection into the gas chromatograph (see D3).

3. Gas Chromatographic Analysis

The concentrated extract of the original water sample, containing the methylated organic acid contaminants, is subjected to gas chromatography at conditions as follows:

Instrument - Gas chromatograph equipped with flame ionization detector (FID) and with temperature programming facility. E.g. Varian Aerograph Model 2700.

Column - 6 ft. x 1/8 in. I.D.; stainless steel; 10% SP216 on Superlcoport; 100/120 mesh.

Column Temperature- 80°C to 185°C programmed at 15°C/min.

Detector Temperature- 300°C

Injector Temperature- 300°C



Carrier Gas - Nitrogen, flow rate 35 ml/min.

Hydrogen - Flow rate 35 ml/min.

Air - Flow rate 180 ml/min.

Chart Speed - 0.25 in./min.

Injection  
Volume - 5 ul.

The gas chromatograms of methylated fatty and resin acids in a standard solution as well as obtained from an effluent sample (Chip Washer Water) are shown in Figures 1 and 2.

#### 4. Organic Acid Standards

##### a. Reagents Used

The following organic acids were used in the preparation of standard solutions:

##### i. Resin Acids:

Pimaric Acid,  $C_{20}H_{30}O_2$ ;  
M.W. 302

Sandaracopimaric Acid,  
 $C_{20}H_{30}O_2$ ;  
M.W. 302

Levopimaric Acid,  
 $C_{20}H_{30}O_2$ ;  
M.W. 302

Isopimaric Acid,  
 $C_{20}H_{30}O_2$ ;  
M.W. 302

Abietic Acid,  
 $C_{20}H_{30}O_2$ ;  
M.W. 302

Dehydroabietic Acid,  
 $C_{20}H_{28}O_2$ ;  
M.W. 300

ii. Fatty Acids

Caproic Acid,  $\text{CH}_3-(\text{CH}_2)_4-\text{COOH}$ ; M.W. 116

Capric Acid,  $\text{CH}_3-(\text{CH}_2)_8-\text{COOH}$ ; M.W. 172

Lauric Acid,  $\text{CH}_3-(\text{CH}_2)_{10}-\text{COOH}$ ; M.W. 200

Myristic Acid,  $\text{CH}_3-(\text{CH}_2)_{12}-\text{COOH}$ ; M.W. 228

Palmitic Acid,  $\text{CH}_3-(\text{CH}_2)_{14}-\text{COOH}$ ; M.W. 256

Stearic Acid,  $\text{CH}_3-(\text{CH}_2)_{16}-\text{COOH}$ ; M.W. 284

Oleic Acid,  $\text{CH}_3-(\text{CH}_2)_7-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$ ; M.W. 282

Linoleic Acid,  $\text{CH}_3-(\text{CH}_2)_4-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$ ; M.W. 280

Linolenic Acid,  $\text{CH}_3-(\text{CH}_2-\text{CH}=\text{CH})_3-(\text{CH}_2)_7-\text{COOH}$ ; M.W. 278

Ricinoleic Acid,  $\text{CH}_3-(\text{CH}_2)_5-\text{COOH}-\text{CH}_2-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$ ; M.W. 298

Arachidic Acid,  $\text{CH}_3-(\text{CH}_2)_{18}-\text{COOH}$ ; M.W. 313

iii. Aromatic Acids

Benzoic Acid,  $\text{C}_7\text{H}_6\text{O}_2$ ; M.W. 122

Salicylic Acid,  $\text{C}_7\text{H}_6\text{O}_3$ ; M.W. 138

iv. Other Reagents Used in Analytical Method:

Hydrochloric Acid, Concentrated; Reagent grade

Sodium Hydroxide, pellets; Reagent grade.

Potassium Hydroxide, pellets; Reagent grade.

Diethyl Ether, anhydrous; glass-distilled.

Magnesium Sulphate, anhydrous; Reagent grade.

Carbitol (diethyleneglycol moniethyl ether); re-distilled.

p-Tolylsulphonylmethylnitrosamide; C.P.

Glass Fibre Filter Paper; Grade 934AH Reeve

Angel by Whatman Inc.

b. Preparation of Stock Solution

Stock solutions are made up from each fatty acid, at 5000 ppm(wt/v) concentrations, by transferring 250 mg of the respective fatty acid into a 50 ml volumetric flask, adding diethyl ether to dissolve the acid and subsequently making up to volume with diethyl ether.

Stock solutions are made up from each resin acid, at 10,000 ppm (wt/v) concentrations, by transferring 500 mg of the respective resin acid into a 50 ml volumetric flask, adding diethyl ether to dissolve the acid and subsequently making up to volume with diethyl ether. These stock solutions are kept in stoppered flasks in the freezer until used for standarization.

c. Preparation of Standard Solutions

A "Working Standard", containing all of the fatty and resin acids, is prepared immediately before use by transferring exactly 400 ml of each of the above stock solutions of fatty and resin acids into a 100 ml volumetric flask and then making up to volume with diethyl ether. This "Working Standard" contains 20 mg of each fatty acid and 40 mg of each resin acid in 100 ml ethereal solution, i.e. 200 ppm (wt/v) and 400 ppm (wt/v), respectively.

A "Standard Solution" is prepared by transferring exactly 1.0 ml of the "Working Solution" into a 15 ml calibrated conical glass tube and subjecting it to methylation by addition of the ethereal solution of diazomethane under the conditions described in 2b. The reaction mixture, brown down to exactly 1.0 ml volume, contains the fatty and resin acids, as their methyl esters, in quantities of 0.2 mg and 0.4 mg, respectively. Aliquots of 5 ul are taken from this "Standard Solution" with a syringe for injection into the gas chromatograph.

d. Calculation

The fatty and resin acid concentrations in the aqueous samples are calculated by relating the respective gas chromatographic peak heights with those obtained from the "Standard Solution".

$$Y = \frac{X}{S} \cdot \frac{m}{v} \cdot \frac{1000}{1}, \text{ where}$$

Y = Concentration (ppm) of fatty or resin acid in sample.

X = Peak height of fatty or resin acid in sample

S = Peak height of fatty or resin acid in "Standard Solution".

m = Fatty or resin acid (mg) in 1 ml of "Standard Solution".

v = Volume of aqueous sample (ml) used in analysis.

5. Recovery Tests

The extraction efficiency and the losses incurred during the concentration procedures of the analytical method were investigated by carrying out recovery tests on "synthetic" aqueous samples containing known amounts of fatty and resin acids.

A "synthetic" sample is prepared as follows: Exactly 2.50 ml of the Working Standard (4c), which contains each of the fatty acids and each of the resin acids at 200 ppm (wt/v) and 400 ppm (wt/v), respectively, is transferred into a 500 ml volumetric flask. After the addition of about 200 ml tap water, the small amount of diethyl ether on the surface is evaporated by passing a stream of nitrogen gas into the flask. The aqueous solution is adjusted to pH3 with concentrated hydrochloric acid and then made up to volume with tap water. The resulting "synthetic" sample of 500 ml, containing each of the fatty acids at 1 ppm (wt/v) concentration and each of the resin acids at 2 ppm (wt/v) concentration, is then subjected to exactly the same sequence of procedures as described in D1 to D3 for the analytical method.

The resulting gas chromatographic peaks obtained from this "synthetic" sample are then directly compared with the corresponding peaks obtained from the methylation product of exactly 2.50 ml undiluted organic acid mixture of the Working Standard. The percent recovery of each fatty and resin acid by the extraction procedure is calculated as follows:

$$Z = \frac{X \cdot 100}{Y}, \text{ where}$$

Z = Percent recovery of fatty or resin acid

X = Peak height of fatty or resin acid in "synthetic" sample.

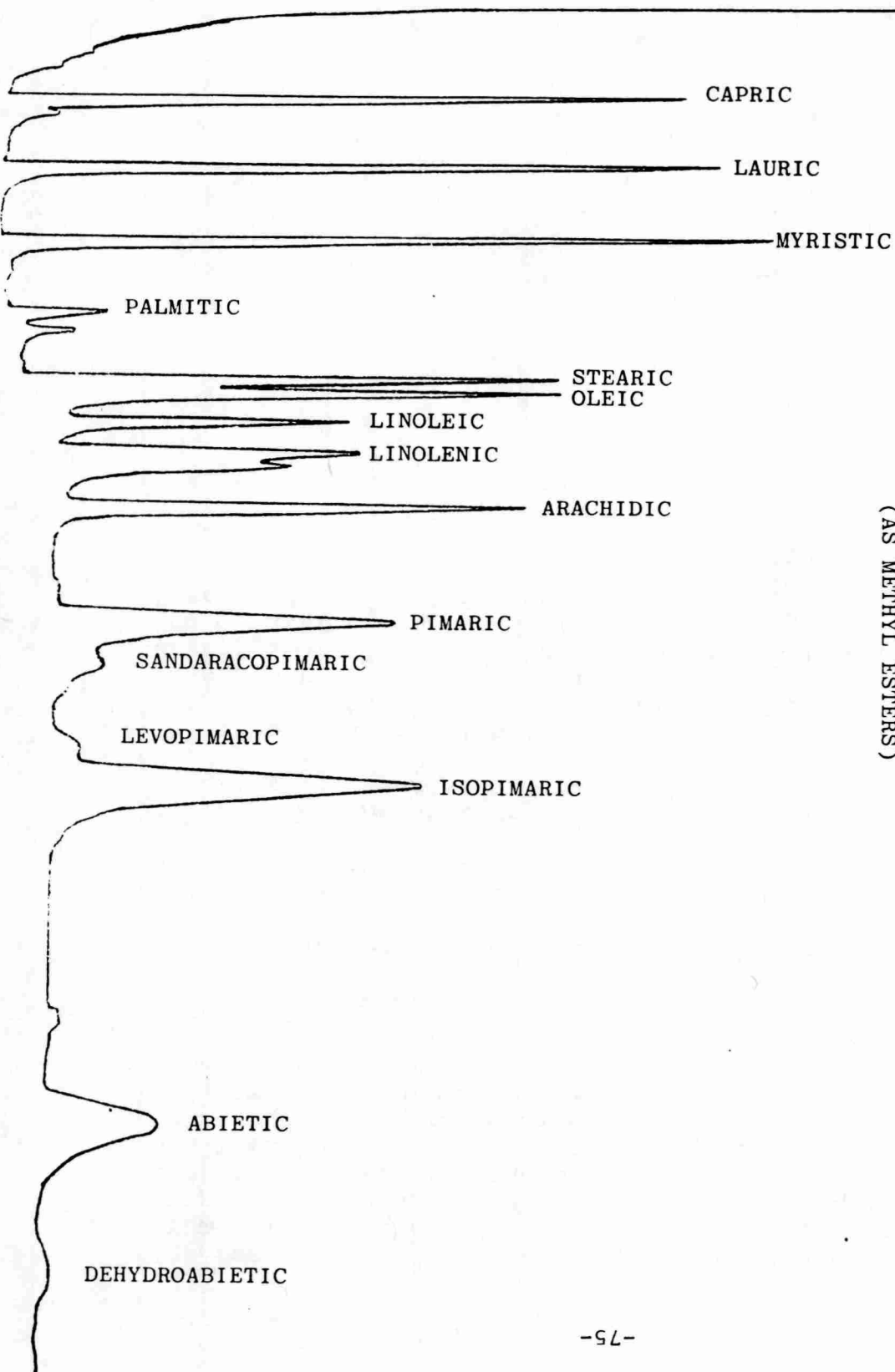
Y = Peak height of fatty or resin acid in "Working Standard" (starting material)

The analytical results from three independent recovery tests are shown in Table 1. They indicate that reasonably high extraction efficiencies of 87% to 97% have been achieved under the conditions of the analytical method for all fatty and resin acids investigated.

APPENDIX III

FIGURE 1

GAS CHROMATOGRAM: FATTY AND RESIN ACIDS IN "STANDARD SOLUTION"  
(AS METHYL ESTERS)

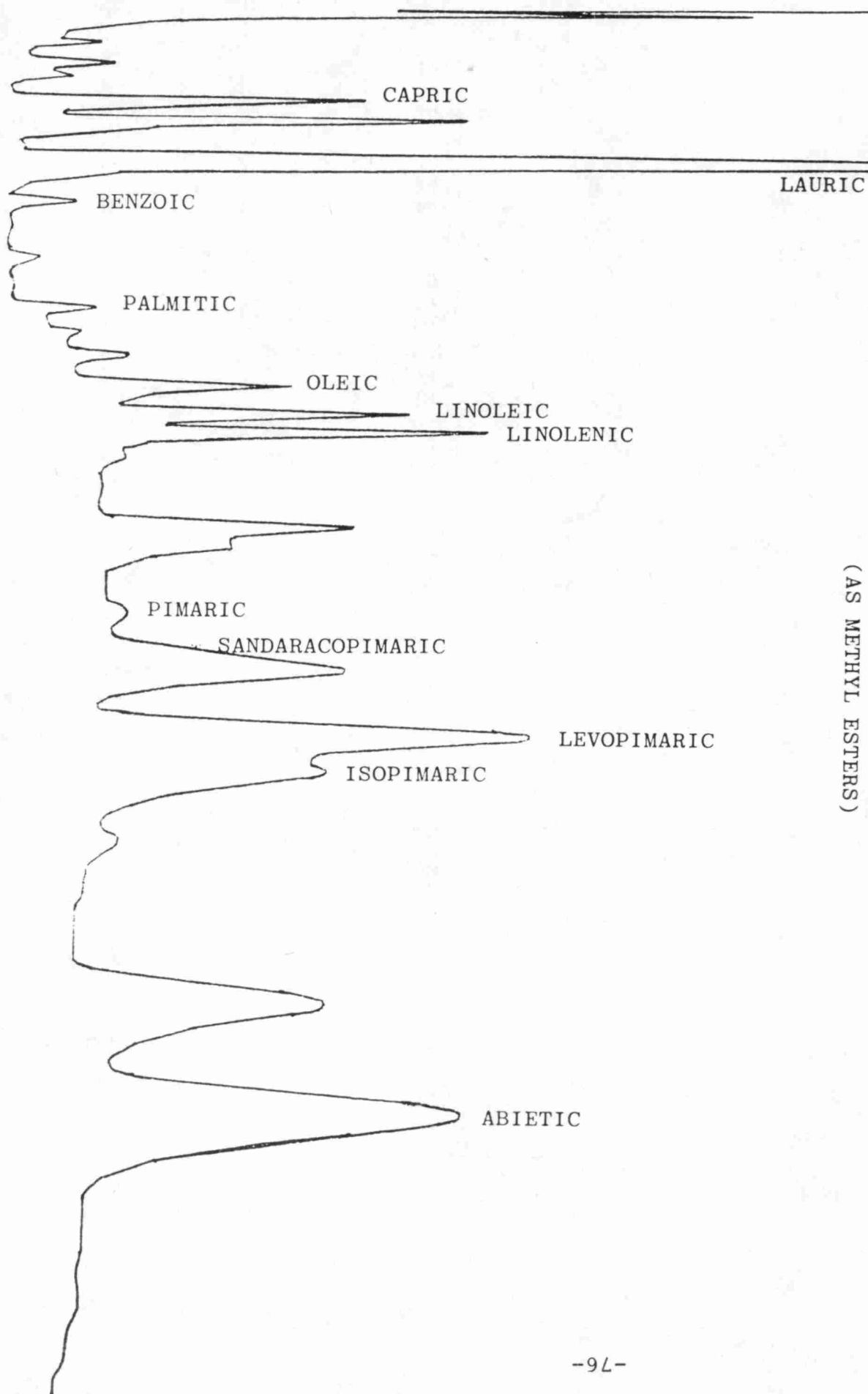


APPENDIX III

FIGURE 2

SAMPLE GAS CHROMATOGRAM: FATTY AND RESIN ACIDS IN OCC53

(AS METHYL ESTERS)



APPENDIX III

TABLE 1

RECOVERIES OF FATTY AND RESIN ACID STANDARDS

BY ANALYTICAL METHOD

<u>Organic Acid</u>	<u>Recovery, %</u>	<u>Mean, %</u>
Capric	93 88 93	91
Lauric	94 94 94	94
Myristic	92 95 95	94
Palmitic	91 95 94	93
Stearic	93 97 94	95
Oleic	94 96 93	94
Linoleic	88 93 88	90
Linolenic	88 88 94	90
Arachidic	89 90 87	89
Isopimaric	94 94 88	92
Abietic	88 90 87	88



APPENDIX III (1)

RESULTS OF RESIN AND FATTY

ACID ANALYSES

1977

APPENDIX III (1)

TABLE 1

TMP WARM RAW WATER

RESIN ACIDS

(all concentrations appear as mg/l)

1977

Lab No.	Date Sampled	Pimaric Acid	Sandaraco Pimaric A	Levopimaric Acid	Isopimaric Acid	Abietic Acid	Dehydro- Abietic A	Total Resin
OCC-16	26/4	trace	trace	trace	0.06	0.09	N.D.	0.15
OCC-44	14/6	N.D.	N.D.	N.D.	N.D.	0.11	trace	0.11
OCC-45	14/6 (10:20 AM)	N.D.	trace	N.D.	N.D.	trace	trace	trace
OCC-50	14/6 (11:45 PM)	N.D.	N.D.	trace	trace	trace	trace	trace
OCC-51	14/6 Duplicate	N.D.	N.D.	trace	trace	trace	trace	trace
OCC-55	14/6 (2:55 PM)	N.D.	trace	trace	trace	trace	N.D.	trace
OCC-56	14/6 (Duplicate)	N.D.	trace	.17	trace	.07	N.D.	trace
OCC-59	15/6 (8:30 AM)	trace	N.D.	N.D.	N.D.	trace	N.D.	trace
AVERAGES		trace	trace	trace	trace	.10	trace	trace - .13

N.A. Data Not Available

N.D Not detected

NOTE: Detection Limits .05 - .07 mg/l for resin acids.

APPENDIX III (i)

TABLE 2

TMP WARM RAW WATER

FATTY ACIDS

(all concentrations appear as mg/l)

1977

Lab No.	Date Sampled	Capric Acid	Lauric Acid	Myristic Acid	Palmitic Acid	Stearic Acid	Lino-leic Acid	Lino-lenic Acid	Arach-idic Acid	Oleic Acid	Total Fatty Acid
OCC-16	16/4	N.D.	N.D.	.04	N.A.	N.A.	.04	N.D.	N.D.	N.A.	.08
OCC-44	14/6 (10:20 AM)	N.D.	N.D.	N.D.	trace	trace	trace	N.D.	N.D.	trace	trace
OCC-45	14/6 (10:20 AM)	N.D.	N.D.	N.D.	trace	trace	trace	N.D.	N.D.	trace	trace
OCC-50	14/6 (11:45 AM)	.05	N.D.	.03	trace	trace	.05	N.D.	N.D.	trace	.13
OCC-51	14/6 Duplicate	.10	N.D.	.05	trace	trace	trace	N.D.	N.D.	trace	.15
OCC-55	14/6 (2:55 P.M.)	.09	.70	N.D.	trace	N.D.	trace	N.D.	N.D.	trace	.79
OCC-56	14/6 (Duplicate)	trace	.64	trace	trace	N.D.	trace	trace	N.D.	trace	.64
OCC-59	15/6 (8:30 AM)	.06	trace	N.D.	N.D.	N.D.	trace	trace	N.D.	N.D.	trace
AVERAGES		trace - .08	trace - .67	.03	trace	trace	.04	trace	trace	trace	trace - .36

N.A. Data Not Available

N.D. Not detected

NOTE: Detection Limit .02 mg/l for fatty acids.

APPENDIX III (i)  
TABLE 3  
TMP CHIP WASHER WATER  
RESIN ACIDS  
(all concentrations appear as mg/l)  
1977

Lab No.	Date Sampled	Pimaric Acid	sandaraco Pimaric A	Levopimaric Acid	Isopimaric Acid	Abietic Acid	Dehydro-Abietic A	Total Resin
OCC-17	26/4	0.74	1.89	6.37	3.18	93.15	29.20	134.43
OCC-40	14/6	0.87	1.32	N.A.	3.31	51.07	11.16	67.73
OCC-41	14/6 Duplicate	1.07	1.36	N.A.	6.01	90.30	7.89	106.63
OCC-46	14/6 (11:35 AM)	N.A.	1.37	N.A.	5.07	87.57	11.02	105.03
OCC-47	14/6 Duplicate	N.A.	1.14	N.A.	4.52	78.13	8.35	92.14
OCC-53	14/6 (2:50 PM)	1.16	N.A.	70.94 (1)	4.51	10.54	N.A.	109.9
OCC-54	14/6 Duplicate	1.69	N.A.	66.48 (1)	4.87	99.7	N.A.	106.3
OCC-60	15/6 (8:30 AM)	1.33	N.A.	10.56	2.53	58.15	3.67	76.24
AVERAGE		1.14	1.42	8.47	4.25	82.9	11.9	99.2

(1) Data appear uncharacteristic. Omitted from average

(2) NOTE: Detection limits .05 - .07 mg/l for resin acids.

APPENDIX III (i)

TABLE 4

TMP CHIP WASHER WATER

FATTY ACIDS

(all concentrations appear as mg/l)

1977

Lab No.	Date Sampled	Capric Acid	Lauric Acid	Myristic Acid	Palmitic Acid	Stearic Acid	Lino-leic Acid	Lino-lenic Acid	Arach-idic Acid	Oleic Acid	Total Fatty Acid
OCC-17	26/4	1.68	5.58	.16	N.A.	N.A.	1.57	1.24	5.96	N.A.	16.19
OCC-49	14/640 (10:10 AM)	1.37	.25	.25	-	-	4.28	N.A.	6.18	-	12.33
OCC-41	14/6 Duplicate	N.A.	.32	.31	-	-	4.88	N.A.	4.89	-	10.40
OCC-46	14/6 (11:35 AM)	N.A.	N.A.	.93	-	-	5.05	N.A.	4.19	-	10.17
OCC-47	14/6 (Duplicate)	N.A.	N.A.	.41	-	-	4.34	N.A.	3.56	-	8.31
OCC-53	14/6 (2:50 P.M.)	.10	32.29 (1)	N.D.	.88	N.D.	2.81	N.D.	N.D.	1.17	4.92
OCC-54	14/6 (Duplicate)	.10	29.10 (1)	N.D.	.81	N.D.	3.45	N.D.	N.D.	1.47	5.83
OCC-60	15/6 (8:30 AM)	trace	2.94	N.D.	.55	N.D.	2.16	N.D.	N.D.	.97	6.62
	AVERAGES	.81	2.27	.41	.75	N.D.	3.58	N.D.	4.96	1.20	9.35

(1) Data appear uncharacteristic: Omitted from weekly averages

N.A. Data not available

N.D. Not detected

NOTE: Detection Limits .02 - .07 mg/l for fatty acids

APPENDIX III (i)

TABLE 5

TMP FOURTH STAGE CLEANER REJECTS

RESIN ACIDS

(all concentrations appear as mg/l)

1977

Lab No.	Date Sampled	Pimaric Acid	Sandaraco Pimaric A	Levopimaric Acid	Isopimaric Acid	Abietic Acid	Dehydro-Abietic A	Total Resin Acid
OCC-57	14/6	.35	N.A.	15.77	1.12	33.15	N.A.	50.39
OCC-55	(2:50 PM)	.35	N.A.	8.45	1.01	36.09	N.A.	45.96
OCC-61	15/6 (8:30 AM)	.22	.90	7.38	1.04	30.54	3.86	45.95
OCC-18	26/4	.56	.40	2.18	1.98	69.77	11.20 (1)	86.09
OCC-42	14/6 (10:15 AM)	.51	.97	3.71	1.57	32.74	2.76	42.26
OCC-43	Duplicate	.64	1.00	3.55	1.62	32.35	1.29	40.45
OCC-48	16/4 Duplicate	.47	.48	N.A.	.62	29.64	trace	31.21
OCC-49	Duplicate	.90	1.75	6.29	1.08	15.40	.58	26.00
AVERAGE		.50	.92	6.76	1.26	34.96	2.12	46.03

(1) Data appears uncharacteristic. Omitted from averages

NOTE: Detection Limits .05 - .07 mg/l for resin acids.

## APPENDIX III (i)

TABLE 6

## TMP FOURTH STAGE CLEANER REJECTS

## FATTY ACIDS

(all concentrations appear as mg/l)

1977

Lab No.	Date Sampled	Capric Acid	Lauric Acid	Myristic Acid	Palmitic Acid	Stearic Acid	Linoleic Acid	Linolenic Acid	Arachidic Acid	Oleic Acid	Total Fatty Acid
OCC-18	26/4	1.55	.62	.06	N.A.	N.A.	1.64	.77	1.09	N.A.	5.73
OCC-42	14/6 (10:15 AM)	.56	.86	.13	-	.30	2.64	.18	1.76	.75	7.18
OCC-43	Duplicate	.53	.89	.12	-	.30	2.62	.19	2.03	.53	7.21
OCC-48	14/6 Duplicate	trace	N.D.	N.D.	-	.30	1.93	.07	1.43	.38	4.11
OCC-49	Duplicate	trace	N.D.	trace	-	(1).0162	trace	trace	.48	.61	1.09
OCC-57	14/6 (2:50 pm)	trace	.42	N.D.	.88	N.D.	1.32	N.D.	N.D.	.47	3.09
OCC-58	14/6	trace	.42	N.D.	.83	N.D.	1.47	N.D.	N.D.	.47	3.19
OCC-61	15/6 (8:30 am)	N.D.	.40	N.D.	.86	trace	1.43	N.D.	N.D.	.45	3.14
AVERAGES		trace -.88	.60	trace -.10	.86	N.D. -.30	1.86	.30	1.34	.52	4.34

(1) Data Appears uncharacteristic: Omitted from averages

N.A. Data Not Available

N.D. Not Detected

NOTE: Detection Limits .02 mg/l for fatty acids.

APPENDIX III (i)

TABLE 7

TMP STOCK LIQUOR - (NO BLEACH ADDED)

RESIN ACIDS

1977

<u>Lab No.</u>	<u>Date Sampled</u>	<u>Pimaric Acid</u>	<u>Sandaraco Pimaric A.</u>	<u>Levopimaric Acid</u>	<u>Isopimaric Acid</u>	<u>Abietic Acid</u>	<u>Dehydro- Abietic A.</u>	<u>Total Resin</u>
OCC-62	15/6 (1:15 PM)	0.37	N.A.	1.56	0.79	23.26	trace	25.98
OCC-63	15/6 (Duplicate	0.47	N.A.	2.21	0.85	25.68	trace	29.21
AVERAGE		0.42	N.A.	1.89	0.82	24.47	trace	27.60

TMP STOCK LIQUOR - (WITH BLEACH ADDED)

OCC-64	15/6 (2:30 PM)	0.37	0.49	1.67	0.87	25.52	6.69	35.61
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N. A. - Not Available

NOTE: - Detection Limits .05 - .07 mg/l for resin acids



APPENDIX III (i)

TABLE 8

TMP STOCK LIQUOR - (NO BLEACH ADDED)

FATTY ACIDS

1977

Lab No.	Date Sampled	Capric Acid	Lauric Acid	Myristic Acid	Palmitic Acid	Stearic Acid	Lino- leic A.	Lino- lenic A.	Arachidic Acid	Oleic Acid	Total Fatty A.
OCC-62	15/6 (1:15 PM)	N.D.	0.24	N.D.	0.21	N.D.	0.98	N.D.	N.D.	0.47	1.90
OCC-63	15/6 Duplicate	N.D.	0.25	N.D.	0.30	N.D.	0.85	N.D.	N.D.	0.41	1.81
	AVERAGES	N.D.	0.24	N.D.	0.26	N.D.	0.92	N.D.	N.D.	0.44	0.86

TMP STOCK LIQUOR (WITH BLEACH ADDED)

OCC-64	15/6 (2:30 PM)	N.D.	0.19	N.D.	0.24	N.D.	1.37	N.D.	N.D.	0.56	2.36
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N.D.: NOT DETECTED

APPENDIX IV

WATER BALANCE

DATA & CALCULATIONS

APPENDIX IV

TABLE 1

TMP MILL WATER BALANCE - July 13, 1978

(All Flows as Water (1))

<u>Time</u>	<u>Chip Integrator</u> <u>Reading</u> <u>(Counts)</u>	<u>Fresh Water</u> <u>Reading</u> <u>(Counts)</u>	<u>Steam</u> <u>Flow</u> <u>(Calculated)</u>	<u>Stock</u> <u>Reading</u> <u>(Counts)</u>	<u>Sewer</u> <u>Head</u> <u>(inches)</u>
8:06	442884	699881		624459	
8:25					6-7/8
8:40					7-1/2
8:55					7
9:10					7-1/2
9:21	44322	702165		625086	
9:25					7-1/2
Counts	341	2284			

Average flume height: 7-1/4

Corres. 481 lb/min. 1412 USGM 2 USGM 1079 USGM 418 USGM  
Flow: 219 kg/min. 5334 l/min. 8 l/min 4080 l/min 1582 l/min.

Water in chips =  $481 \frac{\text{lb}}{\text{min}} \times \frac{.437 \text{ lb water}}{1 \text{ lb chips}} \times \frac{1 \text{ US gal.}}{8.337 \text{ lb.}}$

= 25 US gal/min. (95 l/min.)

TOTAL WATER INPUT:

Total Input = chip moisture + fresh water + steam condensate  
= 25 + 1412 + 2  
= 1439 USGM (5437 l/min)

TOTAL WATER OUTPUT:

Total Output: = (Stock liquor x  $\frac{100 - \text{consistency}}{100}$ ) + Sewer losses  
= (1079 x .964) + 418  
= (1040 + 418)  
= 1439 USGM (5518 l/min)

WATER BALANCE:

Water Balance =  $\frac{\text{Total Water Output}}{\text{Total Water Input}} \times 100$

=  $\frac{1458}{1439} \times 100$

= 101.3%

APPENDIX II

TABLE 2

TMP MILL WATER BALANCE - July 14, 1978

(All Flows as Water (1))

Time	Chip Integrator Reading (Counts)	Fresh Water Reading (Counts)	Steam Flow (Calculated)	Stock Reading (Counts)	Sewer Head (Inches)
12:06	447392	729526		631435	
13:00					5½
13:15					4½
13:50					5
14:15					4½
14:50					5
15:00					4-1/8
15:15					5
15:47	<u>448451</u>	<u>736706</u>		<u>633642</u>	
Counts	1059	7180		2207	
Average flume height:					4-3/4
Corresp.	532 lb/min.	1505 USGM	2 USGM	1289 USGM	212 USGM
Flow:	242 lb/min.	5704 l/min.	8 l/min.	4705 l/min.	802 l/min.

Water in Chips:

$$= 532 \frac{\text{lb}}{\text{min.}} \times \frac{.437 \text{ lb Water}}{1 \text{ lb Chips}} \times \frac{1 \text{ US gal}}{8.337 \text{ lbs.}}$$

$$= 27 \text{ USGM (102 l/min.)}$$

TOTAL WATER INPUT:

$$\begin{aligned} \text{Total Input} &= \text{chip moisture} + \text{fresh water} + \text{steam condensate} \\ &= 27 + 1507 + 2 \\ &= 1536 \text{ USGM (5814 l/min.)} \end{aligned}$$

TOTAL WATER OUTPUT:

$$\begin{aligned} \text{Total Output} &= (\text{Stock liquor} \times \frac{100 - \text{consistency}}{100}) + \text{sewer losses} \\ &= (1289 \times .964) + 212 \\ &= 1243 + 212 \\ &= 1455 \text{ USGM (5507 l/min.)} \end{aligned}$$

WATER BALANCE:

$$\begin{aligned} \text{Balance} &= \frac{\text{Total Water Output}}{\text{Total Water Input}} \times 100 \\ &= \frac{1455}{1536} \times 100 \\ &= \underline{\underline{94.7\%}} \end{aligned}$$

APPENDIX II

TABLE 3

TMP MILL WATER BALANCE - July 15, 1978

(All Flows as Water (1))

Time	Chip Integrator Reading (counts)	Fresh Water Reading (counts)	Steam Flow (calculated)	Stock Reading (counts)	Sewer Head (inches)
9:16	453541	771448		645516	
9:20					4-3/4
9:45					4-7/8
10:15					4-7/8
10:30					4-7/8
11:15					4-7/8
11:30					4-7/8
11:50					5
12:45					4-7/8
14:04					5-1/4
14:45					4-3/4
15:00					4-3/4
15:25					3-3/4
16:23					4-3/4
16:40					4-3/4
17:35	455916	787188		650586	
Counts:	2375	15740		5070	
Average flume height:					4-3/4
Corr. Flow:	504 lb/min	1463 USGM	2	1312 USGM	212 USGM
	230 kg/min	5537 l/min.	8 l/min	4961 l/min.	801 l/min.
Water in Chips: $504 \frac{\text{lb}}{\text{min}} \times \frac{.437 \text{ lb Water}}{1 \text{ lb chips}} \times \frac{1 \text{ US gal}}{8.337 \text{ lb}}$					
= 26 USGM (98 l/min.)					

TOTAL WATER INPUT:

Total Input: = chip moisture + fresh water + steam condensate  
= 16 + 1463 + 2  
= 1491 USGM (5643 l/min.)

TOTAL WATER OUTPUT:

Total Output = (Stock liquor x  $\frac{100 - \text{consistency}}{100}$ ) + Sewer losses  
= (1312 x .964) + 212  
= 1477 USGM (5586 l/min.)

WATER BALANCE:

Water Balance =  $\frac{\text{Total Water Output} \times 100}{\text{Total Water Input}}$   
=  $\frac{1477 \times 100}{1491}$   
= 99.1%

APPENDIX II

TABLE 4

TMP Mill Water Balance - July 16, 1978

(All Flows as Water (1))

<u>Time</u>	<u>Chip Integrator</u> <u>Reading</u> <u>(counts)</u>	<u>Fresh Water</u> <u>Reading</u> <u>(counts)</u>	<u>Steam</u> <u>Flow</u> <u>(calculated)</u>	<u>Stock</u> <u>Reading</u> <u>(counts)</u>	<u>Sewer</u> <u>Head</u> <u>(inches)</u>
9:13	460588	818783		661490	5
9:35					5½
10:12					4-3/4
11:05					5
11:35					5
15:30					6-7/8
15:45					5
15:55					4-3/4
16:05					4-3/4
16:50	<u>462871</u>	<u>834480</u>	<u>          </u>	<u>666795</u>	<u>          </u>
Counts:	2283	15697		5305	
Average flume height:					5¼
Corr. flow:	529 lb/min 240 kg/min	1593 USGM 6029 l/min.	2 8 l/min.	1449 USGM 5480 l/min.	249 USGM 942 l/min.

$$\begin{aligned} \text{Water in Chips: } & 529 \frac{\text{lb}}{\text{min}} \times \frac{.437 \text{ lb Water}}{1 \text{ lb chips}} \times \frac{1 \text{ US gal.}}{8.337 \text{ lb.}} \\ & = 28 \text{ USGM (106 l/min.)} \end{aligned}$$

TOTAL WATER INPUT:

$$\begin{aligned} \text{Total Water Input:} &= \text{chip moisture} + \text{fresh water} + \text{steam condensate} \\ &= 28 + 1593 + 2 \\ &= 1623 \text{ USGM (6143 l/min.)} \end{aligned}$$

TOTAL WATER OUTPUT:

$$\begin{aligned} \text{Total Water Output:} &= (\text{Stock liquor} \times \frac{100 - \text{consistency}}{100}) + \text{sewer losses} \\ &= (1499 \times 0.964) + 249 \\ &= 1694 \text{ USGM (6412 l/min.)} \end{aligned}$$

WATER BALANCE:

$$\begin{aligned} \text{Water Balance} &= \frac{\text{Total Water Output} \times 100}{\text{Total Water Input}} \\ &= \frac{6412}{6143} \times 100 \\ &= 104.4\% \end{aligned}$$

APPENDIX V

SUMMARY OF SAMPLE CONCENTRATIONS

AND WASTE GENERATION

1978 SURVEY

# APPENDIX V

## TABLE I

### TMP PROCESS WARM WATER

#### SUMMARY OF CONCENTRATIONS AND WASTE GENERATION

Sample No.	Date	BOD5			SUSPENDED SOLIDS			DISSOLVED SOLIDS			pH	FLOW				
		(mg/l)	(1)	(lb/d) (2)	(kg/d) (3)	(mg/l)	(lb/d)	(kg/d)	(mg/l)	(lb/d)		(kg/d)	USGM (4)	m <sup>3</sup> /d (5)		
78-540	July 14/78	32		578		262		6	108	49	190	3440	1560	6.8	1507	8203
-541	(4:00-6:00 pm)	<u>23</u>		<u>416</u>		<u>189</u>		<u>11</u>	<u>198</u>	<u>90</u>	<u>175</u>	<u>3160</u>	<u>1433</u>	<u>6.9</u>		
	Average	27		497		225		8.5	153	69	182	3300	3300		1507	8203
78-604	July 15/78	50		878		398		30	522	236	145	2540	1152	6.3	1463	7963
-605	(8:30 am)	50		878		398		55	962	436	135	2380	1080	6.4		
-606		13		228		103		6	114	52	95	1680	762	6.5		
-607	(2:30 pm)	<u>13</u>		<u>228</u>		<u>103</u>		<u>5</u>	<u>88</u>	<u>40</u>	<u>95</u>	<u>1680</u>	<u>762</u>	<u>6.8</u>		
	Average	31		553		250		25	421	191	117	2070	939		1463	7963
78-683	July 15-16/78	22		420		191		20	382	173	135	2580	1150	6.5	1593	8671
-685	July 16/78	<u>15</u>		<u>286</u>		<u>130</u>		<u>15</u>	<u>286</u>	<u>130</u>	<u>130</u>	<u>2480</u>	<u>1125</u>	<u>6.7</u>		
		<u>18</u>		<u>353</u>		<u>160</u>		<u>17</u>	<u>334</u>	<u>151</u>	<u>132</u>	<u>2530</u>	<u>1147</u>		<u>1593</u>	<u>8671</u>
Average	July 14-16	25		468		212		17	303	137	144	2633	1194		1521	8279

(1) Milligrams per litre (bone dry basis)

(2) Pounds per day (bone dry basis)

(3) Kilograms per day (Bone dry basis)

(4) U.S. gallons per minute; based upon average calibrated input water integrator readings  
Determined empirically in the field.

(5) Cubic meters per day; based upon average calibrated input water integrator readings  
Determined in the field.



APPENDIX V

TABLE 2

CHIP WASHER EFFLUENT

SUMMARY OF CONCENTRATION AND WASTE GENERATION IN EFFLUENTS

Sample No.	Date	BOD <sub>5</sub>			SUSPENDED SOLIDS (1)			DISSOLVED SOLIDS			pH	FLOW	
		(mg/l) (2)	(lb/d) (3)	(kg/d) (4)	(mg/l) (5)	(lb/d) (6)	(kg/d) (7)	(mg/l) (8)	(lb/d) (9)	(kg/d) (10)		USGM (11)	m <sup>3</sup> /d (12)
78-503	July 14/78	1800	2012	913	450	504	228	2190	2450	1111	5.3	93	506
-505		1750	1956	887	615	688	312	2130	2382	1080	5.3		
-507		2250	2516	1141	4230	4730	2145	2610	2918	1324	5.8		
-509		1800	2020	916	1945	2174	987	2760	3086	1399	5.4		
-510		2150	2400	1089	3600	4024	1825	2800	3130	1420	5.8		
-511		2200	2460	1116	4995	5580	2531	2710	3030	1374	6.0		
-513		1550	1720	780	260	290	131	1560	1740	789	6.1		
-554	(12:30 am)	<u>2050</u>	<u>2292</u>	<u>1040</u>	<u>1440</u>	<u>1610</u>	<u>730</u>	<u>2660</u>	<u>2974</u>	<u>1349</u>	5.3	—	—
	July 15/78												
	Average	1944	2174	986	2192	2450	1111	2428	2713	1231		93	506

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- (1) Suspended solids 100 mesh (U.S. Std. Screen)
- (2) Milligrams per litre (bone dry basis)
- (3) Pounds per day (bone dry basis)
- (4) Kilograms per day (bone dry basis)
- (5) U.S. gallons per minute. Based upon average daily flow determined empirically in the field.
- (6) Cubic meters per day. Based upon average daily flow determined empirically in the field.
- (7) Data appear uncharacteristic. Omitted.

# APPENDIX V

## TABLE 3

### CHIP WASHER EFFLUENT

#### SUMMARY OF CONCENTRATION AND WASTE GENERATION IN EFFLUENTS

Sample No.	Date	BOD <sub>5</sub>			SUSPENDED SOLIDS (1)			DISSOLVED SOLIDS			pH	FLOW	
		(mg/l) (2)	(lb/d) (3)	(kg/d) (4)	(mg/l) (5)	(lb/d) (6)	(kg/d) (7)	(mg/l) (8)	(lb/d) (9)	(kg/d) (10)		USGM (11)	m <sup>3</sup> /d (12)
78-565	July 15/78	2400	2794	1267	1695	1872	849	2740	3190	1447	5.2	97	528
-567	(12:30 am)	2300	2678	1215	1780	2072	940	2710	3198	1451	5.1		
-569		2200	2560	1161	1500	1746	792	2730	3070	1393	5.2		
-571		1950	2270	1030	1890	2200	998	2600	3086	1400	5.2		
-573		2300	2678	1215	1750	2038	924	2720	3166	1436	5.2		
-573		2300	2678	1215	1935	2252	1022	2810	3368	1528	5.2		
-583		2050	2396	1087	1725	2018	915	2670	3108	1410	5.2		
-585		2000	2320	1052	1575	1834	832	2640	3072	1393	5.1		
-587		2200	2560	1161	1875	2180	989	2720	3166	1436	5.2		
-589		2200	2560	1161	1415	1648	748	2740	3100	1406	5.3		
-610		2200	2560	1161	1345	1466	665	2520	2934	1331	5.4		
-612		2500	2900	1315	1590	1850	839	2630	3062	1389	5.4		
-614		2300	2678	1215	2440	2840	1288	2700	3142	1425	6.2		
-615	July 16/78	2100	2440	1106	(7)	(7)	(7)	(7)	(7)	(7)	(7)		
	(12:30 am)												
	Average	2214	2578	1169	1732	2016	914	2687	3128	1419		97	528

(1) Suspended solids 100 mesh (U.S. Std. Screen)

(2) Milligrams per litre (bone dry basis)

(3) Pounds per day (bone dry basis)

(4) Kilograms per day (bone dry basis)

(5) U.S. gallons per minute. Based upon average daily flow determined empirically in the field.

(6) Cubic meters per day. Based upon average daily flow determined empirically in the field.

(7) Data appear uncharacteristic. Omitted.

# APPENDIX V

## TABLE 4

### CHIP WASHER EFFLUENT

#### SUMMARY OF CONCENTRATION AND WASTE GENERATION IN EFFLUENTS

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Sample No.	Date	BOD5			SUSPENDED SOLIDS(1)			DISSOLVED SOLIDS			pH	FLOW	
		(mg/l) (2)	(lb/d) (3)	(kg/d) (4)	(mg/l) (1b/d)	(kg/d)	(mg/l) (1b/d)	(kg/d)	(mg/l) (1b/d)	(kg/d)		USGM(5)	m <sup>3</sup> /d (6)
78-616	July 16/78	(7)	(7)	(7)	(7)	(7)	(7)	2850	3146	1427	7.1	92	501
-618	(1:00 am)	2100	2318	1051	2500	2760	1252	2670	2948	1337	5.9		
-620		(7)	(7)	(7)	2330	2462	1116	2550	2816	1277	6.6		
-621		1900	2098	952	1410	1556	706	2510	2772	1257	5.9		
-623		1900	2098	952	1700	1876	851	2585	2854	1257	5.8		
-625		1900	2098	952	1520	1678	761	2600	2870	1295	5.8		
-627		2200	2560	1161	1100	1214	551	2480	2738	1302	5.7		
-629		2000	2208	1002	1455	1606	728	2670	2948	1242	5.9		
-631		1700	2876	851	1550	1712	777	2540	2804	1337	5.7		
-633		2100	2318	1051	1335	1474	669	2450	2704	1272	5.8		
-636	(4:00 pm)	1900	2098	952	1615	1782	808	2460	2716	1232	5.7		
Average		1967	2172	985	1642	1812	822	2579	2848	1292		92	501
Average July 14-16		2042	2308	1047	1855	2093	949	2565	2896	1314		94	512

(1) Suspended solids 100 mesh (U.S. Std. Screen)

(2) Milligrams per litre (bone dry basis)

(3) Pounds per day (bone dry basis)

(4) Kilograms per day (bone dry basis)

(5) U.S. gallons per minute. Based upon average daily flow determined empirically in the field.

(6) Cubic meter per day. Based upon average daily flow determined empirically in the field.

(7) Data appear uncharacteristic. Omitted.

APPENDIX V

TABLE 5

FOURTH STAGE CLEANER REJECTS

SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENTS

Sample No.	Date	BOD <sub>5</sub>			SUSPENDED SOLIDS			DISSOLVED SOLIDS			pH	FLOW	
		(mg/l)	(1) (lb/d)	(2) (kg/d)	(3)	(mg/l)	(1b/d)	(kg/d)	(mg/l)	(1b/d)	(kg/d)	USGM	(4) m <sup>3</sup> /d(5)
78-514	July 14/78	1350	766	347	(6)	5850	2654	(6)	(6)	(6)		48	261
-515	(12:15 am)	1350	766	347	10300	6980	3166	2020	1148	521			
-516		1500	852	386	12300	7220	3275	2020	1148	521			
-518		1400	796	361	12700	5140	2332	2070	1176	533			
-520		1600	908	412	9040	6600	2994	2080	1182	536			
-522		1400	796	361	11600	6200	2812	2100	1192	541			
-549	(11:00 pm)	1500	852	386	10900	6320	2867	1980	1124	510			
Average		1443	820	372	11140	5898	2675	2045	1162	527		48	261

- (1) Milligrams per litre. (bone dry basis)
- (2) Pounds per day (bone dry basis)
- (3) Kilograms per day (bone dry basis)
- (4) U.S. gallons per minute. Based upon daily flows determined empirically in the field.
- (5) Cubic meters per day. Based upon daily flows determined empirically in the field.
- (6) Data appears uncharacteristic. Omitted

APPENDIX V

TABLE 6

FOURTH STAGE CLEANER REJECTS

SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENTS

Sample Date No. _____	BOD <sub>5</sub>			SUSPENDED SOLIDS			DISSOLVED SOLIDS			pH	FLOW	
	(mg/l) (1)	(lb/d) (2)	(kg/d) (3)	(mg/l) (1)	(lb/d) (2)	(kg/d) (3)	(mg/l) (1)	(lb/d) (2)	(kg/d) (3)		USGM (4)	m <sup>3</sup> /d(5)
78-555 July 15/78	1400	796	361	10100	5900	2676	1970	1150	552	5.5	49	267
-577 (9:00 am)	1700	992	450	8980	5240	2377	2070	1208	548	5.2		
-579	1800	1052	477	9760	5700	2586	2130	1244	564	5.1		
-581	1450	846	384	9930	5800	2631	2150	1256	570	5.7		
-608	1700	992	450	10000	5840	2649	2050	1196	543	5.5		
-609 (9:00 pm)	1650	964	437	9640	5644	2560	1930	1128	512	(6)	—	—
Average	1617	944	428	9735	5686	2579	2050	1198	543		49	267

(1) Milligrams per litre. (Bone dry basis)

(2) Pounds per day (bone dry basis)

(3) Kilograms per day (bone dry basis)

(4) U.S. gallons per minute. Based upon daily flows determined empirically in the field.

(5) Cubic meters per day. Based upon daily flows determined empirically in the field.

(6) Data appears uncharacteristic. Omitted.

APPENDIX V

TABLE 7

FOURTH STAGE CLEANER REJECTS

SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENTS

Sample No.	Date	BOD <sub>5</sub>			SUSPENDED SOLIDS			DISSOLVED SOLIDS			pH	FLOW					
		(mg/l)	(1)	(lb/d) (2)	(kg/d) (3)	(mg/l)	(lb/d)	(kg/d)	(mg/l)	(lb/d)		(kg/d)	USGM	(4)	m <sup>3</sup> /d (5)		
78-667	July 16/78	1120		650		295		9170	5320	2413		1960	1138	516	5.4	48	261
-669		1200		698		317		9690	5630	2554		1980	1150	522	5.9		
-671		1240		720		327		9750	5664	2569		2020	1174	533	5.8		
-673		1140		640		290		9350	5432	2464		1960	1138	516	5.9		
-675		1220		708		321		10500	6100	2767		1960	1138	516	5.8		
-677		1140		662		300		9640	5600	2540		1910	1100	499	5.8		
-679		1240		720		327		9630	5596	2538		1930	1122	509	5.8		
-681		1160		674		306		9450	5490	2490		1260	732	332	5.8		
	Average	1178		684		310		9648	5606	2543		1873	1088	494		48	261
		1413		816		370		10174	5730	2599		1989	1149	521		48.3	263

(1) Milligrams per litre. (bone dry basis)

(2) Pounds per day (bone dry basis)

(3) Kilograms per day (bone dry basis)

(4) U.S. gallons per minute. Based upon daily flows determined empirically in the field.

(5) Cubic meters per day. Based upon daily flows determined empirically in the field.

(6) Data appears uncharacteristic. Omitted.

APPENDIX V

TABLE 8

TMP MILL FINAL EFFLUENT(1)

SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENT

Sample No.	Date	BOD5			SUSPENDED SOLIDS			DISSOLVED SOLIDS			pH	FLOW	
		(mg/l) (2)	(lb/d) (3)	(kg/d) (4)	mg/l	lb/d	kg/d	mg/l	lb/d	kg/d		USGM(5)	m <sup>3</sup> /d (6)
78-524	July 14/78	(7)			(7)	(7)		(7)			6.2	221	1203
-525	(12:00 am)	1350	3582	1625	(7)	(7)		1720	4566	2071	5.7		
-526		1250	3318	1505	3760	9980	4526	1570	4166	1890	5.8		
-527		1300	3450	1565	3550	9422	4274	1840	4884	2215	5.8		
-528		1400	3716	1686	4090	10854	4923	1780	4724	2143	5.6		
-530		1600	4246	1926	3430	9100	4128	1820	4830	2190	5.6		
-532		1000	2654	1204	3390	8998	4081	1450	3848	1745	6.0		
-536		1250	3318	1505	3560	9450	4287	1840	4884	2215	5.7		
-538		1250	3318	1505	3400	9024	4093	1760	4672	2192	5.7		
-551		1300	3450	1565	3660	9712	4405	1790	4750	2155	5.8		
-552	(1:30 am)	1400	3716	1686	4220	11200	5080	1810	4804	2179	5.6		
	July 15/78												
	Average	1323	3512	1593	3680	9770	4432	1730	4592	2083		221	1203

-100-

(1) TMP Final Effluent is combination of Chip washer effluent. Fourth stafe cleaner rejects; chip washer grit trap discharge pump gland waters and wash up water.

(2) Milligrams per litre (bone dry basis)

(3) Pounds per day (bone dry basis)

(4) Kilograms per day (bone dry basis)

(5) U.S. gallons per minute. Daily average measured at Parshall Flume

(6) Cubic meters per day. Converted from measure USGM at flume

(7) Data appears uncharacteristic. Omitted

APPENDIX V

TABLE 9.

TMP MILL FINAL EFFLUENT (1)

SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENT

Sample No.	Date	BOD <sub>5</sub>			SUSPENDED SOLIDS			DISSOLVED SOLIDS			pH	FLOW	
		(mg/l) (2)	(lb/d) (3)	(kg/d) (4)	mg/l	lb/d	kg/d	mg/l	lb/d	kg/d		USGM (5)	m <sup>3</sup> /d (6)
78-557	July 15/78	1650	4280	1941	3630	9416	4271	1910	4954	2247	5.7	216	1175
-559	(1:30 am)	1500	3892	1765	3280	8508	3860	1930	5006	2270	5.6		
-561		1600	4150	1882	3760	9754	4424	1990	5162	2341	5.7		
-563		1400	3632	1647	3900	10116	4589	1790	4644	2107	5.8		
-567		1140	2958	1342	2820	7316	3319	1750	4540	2059	6.1		
-569		1100	2854	1295	3020	7834	3554	1880	4876	2211	5.9		
-541		1060	2750	1247	2870	7440	3375	1580	4098	1859	6.3		
-543		(7)	(7)	(7)	3070	7964	3612	2090	5422	2459	6.5		
-545		920	2386	1082	2330	6044	2742	1570	4072	1847	5.9		
-547		1200	3112	1412	2810	7290	3307	1860	4824	2188	6.1		
-549	(12:30 am) July 16/78	<u>1060</u>	<u>2750</u>	<u>1247</u>	<u>3340</u>	<u>8664</u>	<u>3930</u>	<u>1900</u>	<u>4928</u>	<u>2235</u>	<u>6.3</u>		
Average		1263	3276	1486	3160	8212	3725	1841	4776	2166		216	1175

(1) TMP Final Effluent is combination of chip washer effluent, fourth stage cleaner rejects, chip washer grit trap discharge pump gland waters and wash up water.

(2) Milligrams per litre (bone dry basis)

(3) Pounds per day (bone dry basis)

(4) Kilograms per day (bone dry basis)

(5) U.S. gallons per minute. Daily average measured at Parshall Flume.

(6) Cubic meters per day. Converted from measure USGM at flume

(7) Data appears uncharacteristic. Omitted.



APPENDIX V

TABLE 10

TMP MILL FINAL EFFLUENT (1)

SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENT

Sample Date No.	BOD <sub>5</sub>			SUSPENDED SOLIDS			DISSOLVED SOLIDS			pH	FLOW	
	(mg/l) (2)	(lb/d) (3)	(kg/d) (4)	mg/l	lb/d	kg/d	mg/l	lb/d	kg/d		USGM (5)	m <sup>3</sup> /d (6)
78-651 July 16/78	1100	3224	1462	3200	9376	4253	1890	5538	2512	6.0	244	1328
-653 (2:30 am)	1080	3160	1433	3410	9992	4532	1880	5508	2498	6.4		
-655	1180	3458	1569	3630	10636	4824	1920	5626	2552	6.2		
-657	1140	3340	1515	2660	7794	3535	1890	5538	2512	6.0		
-659	1060	3106	1409	3500	10256	4652	1950	5714	2592	5.9		
-661	1080	3164	1435	3250	9522	4319	1900	5568	2526	6.0		
-663	820	2402	1090	(7)	(7)	(7)	(7)	(7)	(7)	5.9		
-665	1220	3574	1621	3460	10138	4599	2120	6212	2818	6.3		
-667 (4:00 pm)	960	2812	1276	(7)	(7)	(7)	1740	5098	2312	5.7		
Average	1071	3138	1423	3300	9670	4386	1911	5600	2540		244	1328
July 14-16 Average	1219	3309	1501	3880	9217	4181	1827	4889	2263		227	1236

(1) TMP Final Effluent is combination of chip washer, fourth stage cleaner rejects, chip washer grit trap discharge pump gland waters and wash up water.

(2) Milligrams per litre (bone dry basis)

(3) Pounds per day (bone dry basis)

(4) Kilograms per day (bone dry basis)

(5) U.S. gallons per minute. Daily average measured at Parshall Flume

(6) Cubic meters per day. Converted from measure USGM at flume

(7) Data appears uncharacteristic. Omitted.

# APPENDIX V

## TABLE 11

TMP WHITEWATER(1)

### SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENTS

Sample No.	Date	BOD <sub>5</sub>			DISSOLVED SOLIDS			pH	FLOW	
		(mg/l (2))	(lb/d) (3)	(kg/d) (4)	(mg/l)	(lb/d)	(kg/d)		(USGM) (5)	m <sup>3</sup> /d (6)
78-790	July 14/78	560	8360	3792	895	13360	6060	5.9	1243	6766
-791	(9:30 am)	580	8660	3928	910	13580	6160	5.9		
-792		580	8660	3928	860	12840	5824	5.9		
-793		580	8660	3928	845	12600	5715	5.9		
-794		540	8060	3660	910	13580	6160	5.9		
-795		580	8660	3928	895	13360	6060	6.0		
-796		540	8660	3656	865	12900	5851	6.0		
-797	(3:00 pm)	560	8360	3792	880	13140	5960	6.0		
	July 14/78									
Average		565	8440	3828	883	13180	5978		1243	6766

- (1) TMP Stock Liquors refer to the liquid fraction of the pulp/water slurry being pumped to the paper mill.
- (2) Milligrams per litre (bone dry basis)
- (3) Pounds per day (bone dry basis)
- (4) Kilograms per day (bone dry basis)
- (5) U.S. gallons per minute. Based upon average calibrated stock, booster pump integrator readings; determined empirically in the field. Corrected for consistency of pulp (3.6% BD)
- (6) Cubic meters per day. Based upon average calibrated stock booster pump integrator readings; determined empirically in the field. Corrected for consistency of pulp (3.6% BD).

APPENDIX V

TABLE 12

TMP WHITEWATER (1)

SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENTS

Sample No	Date	BOD <sub>5</sub>				DISSOLVED SOLIDS			pH	FLOW	
		(mg/l) (2)	(lb/d) (3)	(kg/d) (4)		(mg/l) (lb/d) (kg/d)				(USGM) (5)	m <sup>3</sup> /d (6)
78-798	July 15/78	580	8800	3992		845	12820	5815	5.9	1264	6858
-799		560	8500	3856		815	12360	5606	5.9		
-600		640	9720	4409		925	14040	6369	6.0		
-601		640	9720	4409		935	14180	6432	6.2		
-602		620	9400	4264		880	13360	6060	6.0		
-603	(4:45 pm)	600	9100	4128		870	13200	5987	6.0		
Average		607	9200	4173		878	13320	6042		1264	6858

- (1) TMP Stock Liquors refer to the liquid fraction of the pulp/water slurry being pumped to the paper mill.
- (2) Milligrams per litre (bone dry basis)
- (3) Pounds per day (bone dry basis)
- (4) Kilograms per day (bone dry basis)
- (5) U.S. gallons per minute. Based upon average calibrated stock, booster pump integrator readings; determined empirically in the field. Corrected for consistency of pulp (3.6% BD).
- (6) Cubic meters per day. Based upon average calibrated stock booster pump integrator readings; determined empirically in the field. Corrected for consistency of pulp (3.6% BD).

APPENDIX V

TABLE 13

TMP WHITEWATER (1)

SUMMARY OF CONCENTRATIONS AND WASTE GENERATION IN EFFLUENTS

Sample No.	Date	BOD5				DISSOLVED SOLIDS			pH	FLOW	
		(mg/l) (2)	(lb/d) (3)	(kg/d) (4)		(mg/l) (1b/d)	(kg/d)			(USGM) (5)	m <sup>3</sup> /d (6)
78-687	July 16/78	510	8840	4010		880	15260	6922	6.2	1445	7865
-689	(10:00 am)	440	7640	3466		850	14740	6886	6.1		
-691		440	7640	3466		735	12760	5788	5.9		
-693	(3:30 pm)	<u>470</u>	<u>8160</u>	<u>3701</u>		<u>825</u>	<u>14320</u>	<u>6496</u>	5.9		
	Average	465	8080	3665		823	14280	6477		1445	7865
Average July 14-16		<u>546</u>	<u>8573</u>	<u>3889</u>		<u>861</u>	<u>13593</u>	<u>6166</u>		<u>1317</u>	<u>7171</u>

- (1) TMP Stock Liquors refer to the liquid fraction of the pulp/water slurry being pumped to the paper mill.
- (2) Milligrams per litre (bone dry basis)
- (3) Pounds per day (bone dry basis)
- (4) Kilograms per day (bone dry basis)
- (5) U.S. gallons per minute. Based upon average calibrated stock, booster pump integrator readings; determined empirically in the field. Corrected for consistency of pulp (3.6 % BD)
- (6) Cubic meters per day. Based upon average calibrated stock booster pump integrator readings; determined empirically in the field. Corrected for consistency of pulp (3.6% BD).

TABLE 14

SUMMARY OF SAMPLE CONCENTRATIONS - 1978RESIN ACIDS

(ALL CONCENTRATIONS mg/l)

<u>Sample No.</u>	<u>Date</u>	<u>Sample Description</u>	<u>Isopimaric Acid</u>	<u>Pimaric Acid</u>	<u>Sandara CO P.A.</u>	<u>Levopimaric Acid</u>	<u>Abietic Acid</u>	<u>Dehydroabietic Acid</u>	<u>Neoabietic Acid</u>
OCC-109	July 15/78	Chip Wash. Eff. (5:00-6:00 PM)	21.13	6.59	12.95	16.53	178.19	Trace	92.06
-110	July 15/78	7:00-8:00 PM)	15.37	5.34	9.71	10.03	158.98	26.90	90.98
-111	July 15/78	9:00-10:00 PM)	11.88	4.38	8.41	12.45	146.38	20.82	91.30
-114	July 16/78	9:00 AM	28.31	9.05	16.56	20.04	252.43	7.57	134.09
-115	July 16/78	10:00 AM	9.18	3.13	5.90	5.70	141.96	32.77	64.55
-116	July 16/78	11:00 AM	12.98	20.27	9.42	5.77	142.48	35.30	63.93
-117	July 16/78	12:00 AM	12.90	4.40	8.89	13.67	161.52	22.09	90.23
<u>Final Effluent</u>									
OCC-123	July 15/78	10:30 AM	12.88	2.91	5.76	6.30	100.56	21.37	67.09
-126		8:30 PM	7.32	1.90	3.65	2.51	71.26	11.39	43.20
-128	July 16/78	12:30 AM	22.84	6.34	11.52	12.55	174.52	25.79	91.89
-129	July 16/78	2:30 AM	10.63	2.92	5.30	2.53	81.94	16.67	57.57
-131	July 16/78	6:30 AM	10.42	9.39	5.82	7.98	89.27	11.96	56.85
-133	July 16/78	10:30 AM	8.67	1.70	4.77	2.03	128.43	25.82	55.41
-134	July 16/78	12:30 AM	7.71	2.03	3.91	1.59	81.51	17.37	33.42

APPENDIX V

TABLE 15

SUMMARY OF SAMPLE CONCENTRATIONS - 1978

RESIN ACIDS

(ALL CONCENTRATIONS mg/l)

<u>Sample No.</u>	<u>Date</u>	<u>Sample Description</u>	<u>Isopimaric Acid</u>	<u>Pimaric Acid</u>	<u>Sandara co P.A.</u>	<u>Levopimaric Acid</u>	<u>Abietic Acid</u>	<u>Dehydroabietic Acid</u>	<u>Neoabietic Acid</u>
<u>Centri Rejects</u>									
OCC-137	July 16/78	9:00 AM	2.88	1.20	1.80	1.80	55.86	8.90	26.67
-138	July 16/78	10:00 AM	3.55	1.24	2.00	0.72	47.74	14.23	22.62
-139	July 16/78	11:00 AM	4.60	2.70	3.03	2.61	56.44	6.36	32.35
-140	July 16/78	12:00 AM	4.48	0.94	2.55	1.72	86.77	37.12	19.67
<u>Proc. Warm Water</u>									
OCC-145	July 15/78	(4:00 PM)	0.64	Trace	0.13	Trace	5.36	1.14	1.12
-146	July 16/78	4:00 AM	0.90	0.05	0.16	Trace	4.36	1.09	1.14
<u>TMP Whitewater</u>									
OCC-147	July 15/78	(10:00 AM)	2.15	0.70	1.29	0.16	38.11	6.38	15.98
-148	July 16/78	11:30 AM	2.86	0.62	1.56	0.20	48.07	9.32	14.19

APPENDIX V

SUMMARY OF SAMPLE CONCENTRATIONS - 1978

FATTY ACIDS

(ALL CONCENTRATIONS AS mg/l)

TABLE 16

<u>Sample Number</u>	<u>Date</u>	<u>Sample Description</u>	<u>Capric Acid</u>	<u>Lauric Acid</u>	<u>Myristic Acid</u>	<u>Palmitic Acid</u>	<u>Stearic Acid</u>	<u>Oleic Acid</u>	<u>Linoleic Acid</u>	<u>Linolenic Acid</u>	<u>Arachidic Acid</u>	<u>Phthalic Acid</u>	<u>Benzoic Acid</u>
OCC - 109	July 15/78	Chip Washer											
		(5:00-6:00 PM)	N.D.	N.D.	N.D.	N.D.	N.D.	1.32	6.23	N.D.	N.D.	N.D.	8.65
- 110	July 15/78	7:00 PM	N.D.	N.D.	N.D.	N.D.	N.D.	1.13	4.93	N.D.	N.D.	N.D.	17.67
- 111	July 15/78	9:00 PM	N.D.	N.D.	N.D.	N.D.	N.D.	1.61	4.87	N.D.	N.D.	N.D.	44.26
- 114	July 16/78	9:00 AM	N.D.	N.D.	N.D.	N.D.	N.D.	3.76	12.67	N.D.	N.D.	N.D.	20.84
- 115	July 16/78	10:00 AM	N.D.	N.D.	N.D.	N.D.	N.D.	1.93	5.65	N.D.	N.D.	N.D.	5.29
- 116	July 16/78	11:00 AM	N.D.	N.D.	N.D.	N.D.	N.D.	1.91	5.39	N.D.	N.D.	N.D.	6.43
- 117	July 16/78	12:00 AM	N.D.	N.D.	N.D.	N.D.	N.D.	2.13	5.82	N.D.	N.D.	N.D.	48.23
OCC - 123	July 15/78	<u>Final Effluent</u>	N.D.	N.D.	N.D.	N.D.	N.D.	0.79	3.18	N.D.	N.D.	N.D.	9.44
- 126	July 16/78	8:30 PM	N.D.	N.D.	N.D.	N.D.	N.D.	0.24	1.02	N.D.	N.D.	N.D.	9.58
- 128	July 16/78	12:30 AM	N.D.	N.D.	N.D.	N.D.	N.D.	2.53	8.41	N.D.	N.D.	N.D.	10.55
- 129	July 16/78	2:30 AM	N.D.	N.D.	N.D.	N.D.	N.D.	0.93	3.24	N.D.	N.D.	N.D.	5.15
- 131	July 16/78	6:30 AM	N.D.	N.D.	N.D.	N.D.	N.D.	1.11	3.94	N.D.	N.D.	N.D.	9.18
- 133	July 16/78	10:30 AM	N.D.	N.D.	N.D.	N.D.	N.D.	1.70	4.84	N.D.	N.D.	N.D.	3.27
- 134	July 16/78	12:30 PM	N.D.	N.D.	N.D.	N.D.	N.D.	1.30	3.06	N.D.	N.D.	N.D.	2.78

N.D. Not Detectable.

(Detection Limit 0.01 mg/l)

# APPENDIX V

## SUMMARY OF SAMPLE CONCENTRATIONS - 1978

### FATTY ACIDS

(ALL CONCENTRATIONS AS mg/l)

TABLE 17

Sample Number	Date		Capric Acid	Lauric Acid	Myristic Acid	Palmitic Acid	Stearic Acid	Oleic Acid	Linoleic Acid	Linolenic Acid	Arachidic Acid	Phthalic Acid	Benzoic Acid
OCC - 137	July 15/78	<u>Centri Rejects</u>											
		9:00 AM	N.D.	N.D.	N.D.	0.32	N.D.	0.47	1.53	N.D.	N.D.	N.D.	0.32
- 138		10:00 AM	N.D.	N.D.	N.D.	0.10	N.D.	0.61	1.76	N.D.	N.D.	N.D.	0.16
- 139	July 16/78	11:00 AM	N.D.	N.D.	N.D.	Trace	N.D.	0.72	2.92	N.D.	N.D.	N.D.	0.44
- 140		12:00 AM	N.D.	N.D.	N.D.	0.39	N.D.	0.58	1.51	N.D.	N.D.	N.D.	0.27
		<u>Process Washer</u>											
OCC - 145	July 15/78	4:00 PM	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.03	N.D.	N.D.	N.D.	0.68
- 146	July 16/78	4:00 AM	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.03	N.D.	N.D.	N.D.	0.61
		<u>TMP Whitewater</u>											
- 147	July 15/78	10:00 AM	N.D.	N.D.	N.D.	0.12	N.D.	0.63	1.47	N.D.	N.D.	N.D.	0.16
- 147	July 16/78	11:30 AM	N.D.	N.D.	N.D.	0.14	N.D.	0.65	1.69	N.D.	N.D.	N.D.	0.31

N.D. = Not Detectable

(Detectable Limit 0.01 mg/l)



APPENDIX

VI

BIOASSAY

TEST RESULTS

TOXICITY TEST REPORT - SUMMARY

1.

Sample No: 78-40 Sample Type: Process Waste Test No: S-83  
 Date Collected: July 15/78 Date Received: July 17/78 Date(s) Tested: July 18/78  
 Sample Name: Spruce Falls Power & Paper Co.  
 Collection Site: Process Warm Water  
 Region: North East Program: Regular  
 Collected By: M. McKenney Collection Time: unknown  
 Sampling Method: Grab Sample  
 Storage/preservation details: Stored in a covered plastic container at 15°C for  
24 hrs.

Test Species: Salmo gairdneri Life Stage: Fry  
 Test Temp: 14-16°C Mean: 16°C Max: 16°C Min: 16°C  
 Static Test ☒ Renewed Solution ☐ Continuous Flow ☐ Other ☐  
 Test Depth: 45 cm. Test Volume: 40 litres. Aerated ☒ Un-aerated ☐

TEST RESULTS

Median Lethal Concentration (LC-50)

Time	Concentration
4 hours	10% mortality
24 hours	at a concentration
48 hours	of 100% v/v
96 hours	after 96 hrs.

Median Survival Time (MST)

Concentration Tested	Time (Hrs)
100%	>96 hrs

Remarks: The undiluted sample of the Process Warm Water was found to be only marginally  
lethal to rainbow trout (10% mortality after 96 hours of exposure to  
the undiluted sample).

Tested by: K. Holtze, H. Clark, G. Beggs

TOXICITY TEST - BIOLOGICAL DATA

Test Material: Process Warm Water Test Number: S-83

Test Species: Salmo gairdneri Common Name: rainbow trout Stock No. RBT78-6

History:

Supplier: Glenwood Trout Hatchery, Ashburn, Ont.

Treatments: none

Disease: none

Mortalities:

Acclimation Details: Held at 8-9.5°C for 12 days. Temp. raised to 15 by 1°C/day.

Held at 15°C for 7 days

Reference Toxicant Data: None

Holding Temperature at Transfer: 15°C Test Temperature at Transfer: 16°C

Organism Size

	N	$\bar{x}$	$\sigma$	Max	Min
Std. Length (cm)	19	3.91	.32	4.6	3.3
Weight (g)	19	.62	.16	.99	.41

Test Volume (l): 40 Number Animals per Vessel: 10

Replications: single

Loading Rate: .16 g/l (static) or  g/l/day (continuous flow)

	Control	D.O. (ppm)	Highest Concentration (%)	D.O. (ppm)	Exposure Duration (Hrs)
Dissolved Oxygen (mg/l) Start	A	10	A 100	9.8	
Finish	B		B		96

Dilution Water: dechlorinated tap water Total Chlorine Residue: 8 µg/l

pH: 7.44 Conductivity: 325 µmhos/cm @ 25°C

Alkalinity: 88 mg/l CaCO<sub>3</sub> Hardness: 133 mg/l CaCO<sub>3</sub>

Chloride: 29.5 mg/l Sulphate: 31.5 mg/l

Ca: 40 Na: 14 K: 1.4 Mg: 8 mg/l

Reported to: S. Munro P. Williams  
EPS Ontario Region Industrial Abatement  
Env. Can. 83 Algonquin Blvd. W.  
135 St. Clair Ave W Timmins P4N 2R4  
Toronto

Date: Aug. 17/78 Approved: K. Holtze

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TOXICITY TEST REPORT - SUMMARY

1

Sample No: 78-41 Sample Type: Process Waste Test No: S-84  
 Date Collected: July 15/78 Date Received: July 17/78 Date(s) Tested: July 17/78  
 Sample Name: Spruce Falls Power & Paper Co.  
 Collection Site: T.M.P. Chip Washer  
 Region: North East Program: Regular  
 Collected By: M. McKenney Collection Time: unknown  
 Sampling Method: Grab Sample  
 Storage/preservation details: Stored in tightly closed plastic containers at 15°C  
for 24 hrs.

Test Species: Salmo gairdneri Life Stage: Fry  
 Test Temp: 14-16°C Mean: 15 Max: 15 Min: 15  
 Static Test ☒ Renewed Solution ☐ Continuous Flow ☐ Other ☐  
 Test Depth: 45 cm. Test Volume: 40 litres. Aerated ☒ Un-aerated ☐

TEST RESULTS

Median Lethal Concentration (LC-50)

Time	Concentration
4 hours	Non Lethal at 2%
24 hours	1.0 < LC50 < 1.5%
48 hours	1.3%
96 hours	0.9%
96 hr confidence interval	upper limit 1.0% lower limit .8%

Median Survival Time (MST)

Concentration Tested	Time (Hrs)
0.1	>96 hrs
0.5	>96 hrs
1.0	68 hrs
1.5	20 hrs
2.0	16 hrs < MST < 24 hrs.

Remarks: The 96-hr LC50 value for rainbow trout exposed to the T.M.P.  
Chip Washer effluent was found to be 0.9% v/v

Tested by: K. Holtze, H. Clark, G. Beggs

TOXICITY TEST - BIOLOGICAL DATA

Test Material: TMP Chip Washer Test Number: S-84

Test Species: Salmo gairdneri Common Name: rainbow trout Stock No. RBT78-6

History:

Supplier: Glenwood Trout Hatchery, Ashburn, Ont.

Treatments: none

Disease: none

Mortalities:

Acclimation Details: Held at 8-9.5°C for 12 days. Temp. raised to 15 by 1°C/day.

Held at 15°C for 7 days

Reference Toxicant Data: None

Holding Temperature at Transfer: 15°C Test Temperature at Transfer: 15

Organism Size

	N	$\bar{x}$	S	Max	Min
Std. Length (cm)	19	4.06	.38	4.6	3.4
Weight (g)	19	.75	.21	1.04	.45

Test Volume (l): 40 Number Animals per Vessel: 10

Replications: duplicate

Loading Rate: .19 g/l (static) or  g/l/day (continuous flow)

	Control	D.O. (ppm)	Highest Concentration (%)	D.O. (ppm)	Exposure Duration (Hrs)
Dissolved Oxygen (mg/l) Start	A	10	A 2	10	
Finish	B		B		

Dilution Water: dechlorinated tap water central lab Toronto Total Chlorine Residue: 8 µg/l

pH: 7.44 Conductivity: 325 µmhos/cm @ 25°C

Alkalinity: 88 mg/l CaCO<sub>3</sub> Hardness: 133 mg/l CaCO<sub>3</sub>

Chloride: 29.5 mg/l Sulphate: 31.5 mg/l

Ca: 40 Na: 14 K: 1.4 Mg: 8 mg/l

Reported to: S. Munro P. Williams  
EPS Ontario Region Industrial Abatement  
Env. Can. 83 Algonquin Blvd. W.  
135 St. Clair Ave W Tirrimins P4N 2P4  
Toronto

Date: Aug. 17/78

Approved: K. Holtze

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TOXICITY TEST REPORT - SUMMARY

1.

Sample No: 78-42 Sample Type: Process Waste Test No: S-85  
Date Collected: July 15/78 Date Received: July 17/78 Date(s) Tested: July 1  
Sample Name: Spruce Falls Power & Paper Co.  
Collection Site: 4th Stage Rejects  
Region: North East Program: Regular  
Collected By: M. McKenney Collection Time: unknown  
Sampling Method: Grab Sample  
Storage/preservation details: Stored in tightly closed plastic pails at  
15°C for 24 hrs.

Test Species: Salmo gairdneri Life Stage: Fry  
Test Temp: 15 Mean: 15.5°C Max: 16°C Min: 15°C  
Static Test ☒ Renewed Solution ☐ Continuous Flow ☐ Other ☐  
Test Depth: 45 cm. Test Volume: 40 litres. Aerated ☒ Un-aerated ☐

TEST RESULTS

Median Lethal Concentration (LC-50)

Time	Concentration
4 hours	Non lethal at 10%
24 hours	5% LC50 <10%
48 hours	5%
96 hours	3.6%
96 hr confidence interval	upper limit - 4.7% lower limit - 2.8%

Median Survival Time (MST)

Concentration Tested	Time (Hrs)
0.1, 0.5, 1.0, 1.5, 2.0%	
5.0%	48 hr.
10%	12 < MST < 24 hrs

Remarks: The 96 hr LC50 value for rainbow trout fry exposed to the  
4th Stage Rejects effluent was 3.6% v/v

Tested by: K. Holtze, H. Clark, G. Beggs

TOXICITY TEST - BIOLOGICAL DATA

Test Material: 4th Stage Rejects Test Number: S-85

Test Species: Salmo gairdneri Common Name: rainbow trout Stock No: 23778-6

History:

Supplier: Glenwood Trout Hatchery, Ashburn, Ont.

Treatments: none

Disease: none

Mortalities:

Acclimation Details: Held at 8-9.5°C for 12 days. Temp. raised to 15 by 1°C/day.

Held at 15°C for 7 days

Reference Toxicant Data: None

Holding Temperature at Transfer: 15°C Test Temperature at Transfer: 15-16°C

Organism Size

	N	$\bar{x}$	$\sigma$	Max	Min
Std. Length (cm)	20	3.91	.38	4.7	3.3
Weight (g)	20	.63	.19	.94	.40

Test Volume (l): 40 l Number Animals per Vessel: 10

Replications: duplicate

Loading Rate: .16 g/l (static) or  g/l/day (continuous flow)

	Control	D.O. (ppm)	Highest Concentration (%)	D.O. (ppm)	Exposure Duration (Hrs)
Dissolved Oxygen (mg/l) Start	A	9.9	A 2	10	
Finish	B		B		96

Dilution Water: dechlorinated tap water Total Chlorine Residue: 3 µg/l

pH: 7.44 Conductivity: 325 µmhos/cm @ 25°C

Alkalinity: 88 mg/l CaCO<sub>3</sub> Hardness: 133 mg/l CaCO<sub>3</sub>

Chloride: 29.5 mg/l Sulphate: 31.5 mg/l

Ca: 40 Na: 14 K: 1.4 Mg: 8 mg/l

Reported to: S. Munro P. Williams

EPS Ontario Region Industrial Abatement

Env. Can. 83 Algonquin Blvd. W.

135 St. Clair Ave W Timmins P4N 2R4

Toronto

Date: Aug. 17/78 Approved: K. Holtze

## TOXICITY TEST REPORT - SUMMARY

1.

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Sample No. 78-43 Sample Type: Process Waste Test No: S-86  
Date Collected: July 15/78 Date Received: July 17/78 Date(s) Tested: July 17/78  
Sample Name: Soroca Falls Power & Paper Co.  
Collection Site: T.M.P. Stock Liquor  
Region: North East Program: Regular  
Collected By: M. McKenney Collection Time: unknown  
Sampling Method: Grab Sample  
Storage/preservation details: Stored in tightly closed plastic containers at 15°C for 24 hrs.

Test Species: Salmo gairdneri Life Stage: Fry  
Test Temp: 14-16°C Mean: 16°C Max: 16°C Min: 16°C  
Static Test ☒ Renewed Solution ☐ Continuous Flow ☐ Other ☐  
Test Depth: 45 cm. Test Volume: 40 litres. Aerated ☒ Un-aerated ☐

## TEST RESULTS

## Median Lethal Concentration (LC-50)

Time	Concentration
4 hours	Non lethal at 5%
24 hours	" "
48 hours	2 <LC50 <5%
96 hours	2.3%
96 hr confidence interval	upper limit 2.8 lower limit 1.9

## Median Survival Time (MST)

Concentration Tested (%)	Time (Hrs)
.5	Non Lethal at 96 hr.
1.0	" " " " "
1.5	>96 hr
2.0	>96 hr
5.0	36 hr

Remarks: The 96 hr LC50 value for rainbow trout exposed to the TMP Stock  
Liquor effluent was found to be 2.3% v/v

Tested by: K. Holtze, H. Clark, G. Beggs



TOXICITY TEST - BIOLOGICAL DATA

Test Material: TNP Stock Test Number: S-86

Test Species: Salmo gairdneri Common Name: rainbow trout Stock No. 23773-6

History:

Supplier: Glenwood Trout Hatchery, Ashburn, Ont.

Treatments: none

Disease: none

Mortalities:

Acclimation Details: Held at 8-9.5°C for 12 days. Temp. raised to 15 by 1°C/day.  
Held at 15°C for 7 days

Reference Toxicant Data: None

Holding Temperature at Transfer: 15°C Test Temperature at Transfer: 16°C

Organism Size

	N	$\bar{x}$	$\sigma$	Max	Min
Std. Length (cm)	20	3.93	.41	4.7	3.2
Weight (g)	20	.62	.25	1.18	.38

Test Volume (l): 40 Number Animals per Vessel: 10

Replications: duplicate

Loading Rate: .16 g/l (static) or  g/l/day (continuous flow)

	Control	D.O. (ppm)	Highest Concentration (%)	D.O. (ppm)	Exposure Duration (Hrs)
Dissolved Oxygen (mg/l) Start	A	10	A 5%	10	
Finish	B		B		96

Dilution Water: dechlorinated tap water Total Chlorine Residue: 8  $\mu\text{g/l}$   
central lab Toronto  
 pH: 7.44 Conductivity: 325  $\mu\text{mhos/cm @ } 25^\circ\text{C}$   
 Alkalinity: 83  $\text{mg/l CaCO}_3$  Hardness: 133  $\text{mg/l CaCO}_3$   
 Chloride: 29.5  $\text{mg/l}$  Sulphate: 31.5  $\text{mg/l}$   
 Ca: 40 Na: 14 K: 1.4 Mg: 8  $\text{mg/l}$

Reported to: S. Munro P. Williams  
EPS Ontario Region Industrial Abatement  
Env. Can. 83 Algonquin Blvd. W.  
135 St. Clair Ave W Timmins P4N 2R4  
Toronto

Date: Aug. 27/78 Approved: K. Holtze

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TOXICITY TEST REPORT - SUMMARY

1.

Sample No: 78-44 Sample Type: Industrial Effluent Test No: S-87  
 Date Collected: July 15/78 Date Received: July 17/78 Date(s) Tested: July 18/78  
 Sample Name: Spruce Falls Power & Paper Co.  
 Collection Site: T.M.P. Final Effluent  
 Region: North East Program: Regular  
 Collected By: M. McKenney Collection Time: unknown  
 Sampling Method: Grab Sample  
 Storage/preservation details: Stored in tightly closed plastic containers at  
15°C for 24 hrs.

Test Species: Salmo gairdneri Life Stage: Fry  
 Test Temp: 15°C Mean: 16°C Max: 16°C Min: 16°C  
 Static Test ☒ Renewed Solution ☐ Continuous Flow ☐ Other ☐  
 Test Depth: 45 cm. Test Volume: 40 litres. Aerated ☒ Unaerated ☐

TEST RESULTS

Median Lethal Concentration (LC-50)

Time	Concentration
4 hours	Non lethal at 5%
24 hours	>5%
48 hours	2<LC50 <5%
96 hours	3.2%
96 hr Lethal Range	upper concentration 5% lower concentration 2%

Median Survival Time (MST)

Concentration Tested	Time (Hrs)
0.5%	Non Lethal
1.0	" "
1.5	" "
2.0	" "
5.0	24 hr <MST <48 hr

Remarks: The 96 hr LC50 value for rainbow trout exposed to the T.M.P. Final  
Effluent was found to be 3.2 % v/v

Tested by: K. Holtze, H. Clark, G. Beggs

TOXICITY TEST - BIOLOGICAL DATA

Test Material: Final Effluent Test Number: S-87

Test Species: Salmo gairdneri Common Name: rainbow trout Stock No. PBT78-6

History:

Supplier: Glenwood Trout Hatchery, Ashburn, Ont.

Treatments: none

Disease: none

Mortalities:

Acclimation Details: Held at 8-9.5°C for 12 days. Temp. raised to 15 by 1°C/day.

Held at 15°C for 7 days

Reference Toxicant Data: None

Holding Temperature at Transfer: 15°C Test Temperature at Transfer: 16°C

Organism Size

	N	$\bar{x}$	$\sigma$	Max	Min
Std. Length (cm)	20	4.1	.95	4.7	3.6
Weight (g)	20	.75	.14	1.0	.52

Test Volume (l): 40 Number Animals per Vessel: 10

Replications: duplicate

Loading Rate: .19 g/l (static) or  g/l/day (continuous flow)

	Control	D.O. (ppm)	Highest Concentration (%)	D.O. (ppm)	Exposure Duration (Hrs)
Dissolved Oxygen (mg/l) Start	A	10.1	A 5	10	
Finish	B		B		96

Dilution Water: dechlorinated tap water central lab Toronto Total Chlorine Residue: 8  $\mu\text{g/l}$

pH: 7.44 Conductivity: 325  $\mu\text{mhos/cm @ 25°C}$

Alkalinity: 83  $\text{mg/l CaCO}_3$  Hardness: 133  $\text{mg/l CaCO}_3$

Chloride: 29.5  $\text{mg/l}$  Sulphate: 31.5  $\text{mg/l}$

Ca: 40 Na: 14 K: 1.4 Mg: 8  $\text{mg/l}$

Reported to: S. Munro P. Williams  
EPS Ontario Region Industrial Abatement  
Env. Can. 83 Algonquin Blvd. W.  
135 St. Clair Ave W Timmins P4N 2R4  
Toronto

Date: Aug. 17/78 Approved: K. Holtze

APPENDIX

VII

ORGANIC ACID

TREATABILITY STUDY

PROCEDURES FOR THE EXTRACTION OF RESIN AND FATTY  
ACIDS FROM TMP EFFLUENTS BY COLUMNAR RESIN EXCHANGES

Toxicant removal was affected by column chromatography. Eight 1.9 cm x 50 cm glass column were run in parallel. Amberlite XAD-7 (Rohm & Haas, Philadelphia, Pennsylvania) a porous polymer resin of intermediate polarity was utilized for the extraction. The chemical and physical properties of the resin are presented below.

Chemical nature	Acrylic Ester
Parasity Volume %	55
True wet density g/cc	1.05
Surface area m <sup>2</sup> /g	450.
Average pore diameter (Angstrams)	90.
Skeletal Density g/cc	1.24
Nominal mesh size	20-50
Exchange capacity	1.1 mg/cc

RESIN REQUIREMENTS

To establish the amount of resin required to successfully extract all the resin and fatty acids from the sample, the following calculations were performed:

$$\frac{(\text{average concentration of acids*})}{\text{Resin capacity}} \times \begin{matrix} \text{(Volume of sample)} = \\ \text{(Number of mls)} \\ \text{(of resin)} \\ \text{(required)} \end{matrix}$$

\*average concentration determined from historical data.

An additional 10% was added as a safety factor.

#### RESIN PRETREATMENT

The resin, as shipped, requires pretreatment to establish its most active state. The following steps were followed, as outlined by R.O. Blosser, 1978:

- 1) Slurry 300 mls of resin with 600 mls of distilled water 6 times, decanting excess solution and fines after each slurry.
- 2) Slurry 150 mls of resin into each of 8 2.2 x 50 cm column fitted with a stopcock and containing a 1 cm glass wool plug at the bottom. A 2 cm glass wool plug is placed on top of the resin.
- 3) Wash each column with 1000 ml of 9/1 ether/methanol at a rate of 10 ml/min.
- 4) Wash each column with 500 ml of methanol at a rate of 10 ml/min.
- 5) Wash each column with 3000 mls of distilled water at a rate of 20 mls/min.

#### COLUMN PACKING

- a) Check columns to insure cleanliness.
- b) Insert stopcock at bottom and place a glass wool plug over stopcock inlet.

- c) Add approximately 2-3 inches of distilled water to column.
  - d) Slurry the resin into the column with a continuous stream of distilled water.
  - e) Fill column to no more than 90% capacity.
  - f) Place glass wool plug over the resin bed. There should be  $\frac{1}{2}$ " of water on top of the bed.
  - g) Add some effluent to the column. Open the stopcock and allow it to enter the bed. Repeat several times.
  - h) Top up column with effluent.
  - i) Pump sample through the delivery manifold to remove all air from the system.
  - j) Stopper delivery tube(s) into column(s) and begin pumping sample.
- Adjust flow to 10 ml/min/80 ml column.

#### SAMPLE TREATMENT

Samples were acidified to pH 3 with dilute  $H_2SO_4$  prior to passage through the resin bed, as acidic conditions are required for the quantitative extraction of resin and fatty acids from pulp mill wastes (R.O. Blosser, 1978). The sample is then pumped through the resin bed at a flow rate of 10 ml/min/80 ml column. Due to sample volume and acid concentration several resin beds were required for complete extraction.

RESULTS:

Chipwasher

The chipwasher effluent was extremely toxic, with a 96 hr LC<sub>50</sub> of 0.9% v/v. LC<sub>50</sub> & MST values indicate this effluent to be the most toxic of those tested for TMP.

Treatment of this chipwasher effluent with XAD-7 resin to remove resin and fatty acids reduced the 96 hr. LC<sub>50</sub> to 2.7% v/v, with a corresponding MST value for 50% v/v = 2.5 hrs.

FOURTH STAGE REJECTS

This discharge had a 96 hr LC<sub>50</sub> of 3.6% v/v. MST data indicated this to be the 2nd most toxic sample tested.

Treatment of the effluent to remove resin and fatty acids reduced the 96 hr LC<sub>50</sub> to 11.8% v/v, a corresponding MST for 50% v/v was 5.8 hrs.

TMP FINAL EFFLUENT

The 96 h LC<sub>50</sub> for the final effluent of the TMP process was 3.2% v/v, an associated MST value showed 5% v/v solution would support rainbow trout for 24-48 hrs.



PROCESS WARM WATER

The bioassay sample was non-lethal to rainbow trout in 100% v/v concentration over 96 hrs.

CONCLUSIONS

The samples of fourth stage rejects discharge and chipwasher discharge, treated by passage through an XAD-7 ion exchange column, showed reduced levels of resin and fatty acids, when analysed. These reductions in resin and fatty acids were reflected by similar reductions in toxicity, as determined through bioassay.

SUMMARY OF TOXICITY RESULTS

	<u>Process</u> <u>Warm Water</u>	<u>Final</u> <u>Effluent</u>	<u>TMP Stock</u> <u>Liquor</u>	<u>Chipwasher</u> <u>Raw Treatment</u>		<u>4th Stage Rejects</u> <u>Raw Treated</u>	
Toxicity (96 hr. LC <sub>50</sub> )	Non Lethal in 100% (v/v)	3.2%	2.3%	0.9%	2.7%	3.6	11.8%
Confidence Interval	-	2 - 5%	1.9-2.8%	0.8-1.0%	2-5%	2.8-4.7%	10-20%
Median Survival Times (hrs)							
0.1%				96 hr.		Non Lethal	
0.5%		Non Lethal	Non Lethal	96 hr.		"	
1.0%		"	"	68 hr.		"	
1.5%		"	96 hr.	20 hr.		"	
2.0%		"	96 hr.	16 MST 24 hrs.	96 hrs.	"	
5.0%		24 MST 48 hr.	35 hr.		8 MST 16 hr.	48 hr.	
10%					2 MST 5 hr.	12 MST 24 hrs.	96 hr.
20%					2 MST 3 hr.		24 MST 33 hr.
100%	96 hr.				2 MST 3 hr.		5.8 hr.

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An evaluation of efficiency  
of the different  
factorial designs  
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